

checked by IT  
6/20/17

# CERTIFICATION

SDG No: FA41854 Laboratory: Accutest, Florida  
Site: BMS, Humacao, PR Matrix: Groundwater

**SUMMARY:** Groundwater samples (Table 1) were collected on the BSMC, Humacao, PR. Samples were taken March 6, 2017 and were analyzed in Accutest Laboratory of Orlando, Florida that reported the data under SDG No.: FA41854. Results were validated using the latest validation guidelines (July, 2015) of the EPA Hazardous Waste Support Section or the QC requirements of the method employed. The analyses performed are shown in Table 1. Individual data review worksheets are enclosed for each target analyte group. The organic data sample summary form shows for analyte results that were qualified.

In summary the results are valid and can be used for decision making purposes.

Table 1. Samples analyzed and analysis performed

SAMPLE ID	SAMPLE DESCRIPTION	MATRIX	ANALYSIS PERFORMED
FA41854-1	OSMW-6S	Groundwater	VOCs; SVOCs; SVOCs (SIM); VPHs; EPHs; Pesticides
FA41854-2	OSMW-6D	Groundwater	VOCs; SVOCs; SVOCs (SIM); VPHs; EPHs; Pesticides
FA41854-2D	OSMW-6D MSD	Groundwater	VOCs; SVOCs; SVOCs (SIM); Pesticides
FA41854-2S	OSMW-6D MS	Groundwater	VOCs; SVOCs; SVOCs (SIM); Pesticides
FA41854-3	TB030617B	AQ – Trip Blank Water	VOCs

Reviewer Name: Rafael Infante  
Chemist License 1888

Signature:

Date:

*Rafael Infante*

April 14, 2017



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## Report of Analysis

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Client Sample ID: OSMW-6S  
 Lab Sample ID: FA41854-1  
 Matrix: AQ - Ground Water  
 Method: SW846 8260C  
 Project: BMSMC, Humacao, PR

Date Sampled: 03/06/17  
 Date Received: 03/08/17  
 Percent Solids: n/a

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	I46110.D	1	03/09/17	WV	n/a	n/a	VI1288
Run #2							

Run #	Purge Volume
Run #1	5.0 ml
Run #2	

CAS No.	Compound	Result	RL	MDL	Units	Q
71-43-2	Benzene	ND	1.0	0.31	ug/l	
67-66-3	Chloroform	ND	1.0	0.30	ug/l	
75-71-8	Dichlorodifluoromethane	ND	2.0	0.50	ug/l	
107-06-2	1,2-Dichloroethane	ND	1.0	0.31	ug/l	
1634-04-4	Methyl Tert Butyl Ether	ND	1.0	0.23	ug/l	
75-85-4	Tert-Amyl Alcohol	ND	20	5.3	ug/l	
75-01-4	Vinyl Chloride	ND	1.0	0.41	ug/l	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
1868-53-7	Dibromofluoromethane	96%		83-118%
17060-07-0	1,2-Dichloroethane-D4	101%		79-125%
2037-26-5	Toluene-D8	99%		85-112%
460-00-4	4-Bromofluorobenzene	101%		83-118%



ND = Not detected MDL = Method Detection Limit  
 RL = Reporting Limit  
 E = Indicates value exceeds calibration range

J = Indicates an estimated value  
 B = Indicates analyte found in associated method blank  
 N = Indicates presumptive evidence of a compound

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## Report of Analysis

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Client Sample ID:	OSMW-6S	Date Sampled:	03/06/17
Lab Sample ID:	FA41854-1	Date Received:	03/08/17
Matrix:	AQ - Ground Water	Percent Solids:	n/a
Method:	SW846 8270D SW846 3510C		
Project:	BMSMC, Humacao, PR		

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	X052882.D	1	03/14/17	NG	03/11/17	OP64132	SX2241
Run #2							

Run #	Initial Volume	Final Volume
Run #1	1050 ml	1.0 ml
Run #2		

CAS No.	Compound	Result	RL	MDL	Units	Q
100-52-7	Benzaldehyde	ND	24	4.8	ug/l	
117-81-7	bis(2-Ethylhexyl)phthalate <sup>a</sup>	ND	4.8	0.95	ug/l	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
4165-60-0	Nitrobenzene-d5	88%		42-108%
321-60-8	2-Fluorobiphenyl	82%		40-106%
1718-51-0	Terphenyl-d14	75%		39-121%

(a) Associated BS recovery outside control limits.



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 B = Indicates analyte found in associated method blank  
 N = Indicates presumptive evidence of a compound

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## Report of Analysis

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Client Sample ID:	OSMW-6S	Date Sampled:	03/06/17
Lab Sample ID:	FA41854-1	Date Received:	03/08/17
Matrix:	AQ - Ground Water	Percent Solids:	n/a
Method:	SW846 8270D BY SIM SW846 3510C		
Project:	BMSMC, Humacao, PR		

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	W098088.D	1	03/17/17	FS	03/11/17	OP64133	SW4359
Run #2	U060372.D	1	03/14/17	NJ	03/11/17	OP64133	SU2649

Run #	Initial Volume	Final Volume
Run #1	1050 ml	1.0 ml
Run #2	1050 ml	1.0 ml

CAS No.	Compound	Result	RL	MDL	Units	Q
56-55-3	Benzo(a)anthracene	ND	0.19	0.038	ug/l	
123-91-1	1,4-Dioxane	0.72 <sup>a</sup>	0.29	0.14	ug/l	
91-20-3	Naphthalene	ND	0.95	0.38	ug/l	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
4165-60-0	Nitrobenzene-d5	65% <sup>b</sup>	80% <sup>b</sup>	42-108%
321-60-8	2-Fluorobiphenyl	82% <sup>b</sup>	70% <sup>b</sup>	40-106%
1718-51-0	Terphenyl-d14	54% <sup>b</sup>	69% <sup>b</sup>	39-121%

(a) Result is from Run# 2

(b) Surrogate recoveries corrected for actual spike amount.



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 RL = Reporting Limit  
 E = Indicates value exceeds calibration range

J = Indicates an estimated value  
 B = Indicates analyte found in associated method blank  
 N = Indicates presumptive evidence of a compound

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## Report of Analysis

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Client Sample ID:	OSMW-6S	Date Sampled:	03/06/17
Lab Sample ID:	FA41854-1	Date Received:	03/08/17
Matrix:	AQ - Ground Water	Percent Solids:	n/a
Method:	MADEP VPH REV 1.1		
Project:	BMSMC, Humacao, PR		

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	UU019272.D	1	03/10/17	AJC	n/a	n/a	GUU1012
Run #2							

Run #	Purge Volume
Run #1	5.0 ml
Run #2	

## MADEP VPH List

CAS No.	Compound	Result	RL	MDL	Units	Q
	C9- C10 Aromatics (Unadj.)	ND	100	35	ug/l	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
460-00-4	BFB	104%		70-130%
460-00-4	BFB	102%		70-130%



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J = Indicates an estimated value  
 B = Indicates analyte found in associated method blank  
 N = Indicates presumptive evidence of a compound

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## Report of Analysis

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Client Sample ID:	OSMW-6S	Date Sampled:	03/06/17
Lab Sample ID:	FA41854-1	Date Received:	03/08/17
Matrix:	AQ - Ground Water	Percent Solids:	n/a
Method:	MADEP EPH REV 1.1 SW846 3510C		
Project:	BMSMC, Humacao, PR		

	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	NN017833.D	1	03/17/17	MG	03/10/17	OP64122	GNN900
Run #2							

	Initial Volume	Final Volume
Run #1	1020 ml	2.0 ml
Run #2		

## MAEPH List

CAS No.	Compound	Result	RL	MDL	Units	Q
	C11-C22 Aromatics (Unadj.)	ND	200	78	ug/l	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
3386-33-2	1-Chlorooctadecane	47%		40-140%
580-13-2	2-Bromonaphthalene	104%		40-140%
84-15-1	o-Terphenyl	73%		40-140%
321-60-8	2-Fluorobiphenyl	101%		40-140%



ND = Not detected    MDL = Method Detection Limit  
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 B = Indicates analyte found in associated method blank  
 N = Indicates presumptive evidence of a compound

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## Report of Analysis

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Client Sample ID: OSMW-6S  
 Lab Sample ID: FA41854-1  
 Matrix: AQ - Ground Water  
 Method: SW846 8081B SW846 3510C  
 Project: BMSMC, Humacao, PR

Date Sampled: 03/06/17  
 Date Received: 03/08/17  
 Percent Solids: n/a

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	KK82213.D	1	03/17/17	MV	03/10/17	OP64131	GKK2635
Run #2							

Run #	Initial Volume	Final Volume
Run #1	1000 ml	5.0 ml
Run #2		

CAS No.	Compound	Result	RL	MDL	Units	Q
60-57-1	Dieldrin	ND	0.010	0.0024	ug/l	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
877-09-8	Tetrachloro-m-xylene	105%		42-127%
2051-24-3	Decachlorobiphenyl	37%		27-127%



ND = Not detected      MDL = Method Detection Limit  
 RL = Reporting Limit  
 E = Indicates value exceeds calibration range

J = Indicates an estimated value  
 B = Indicates analyte found in associated method blank  
 N = Indicates presumptive evidence of a compound

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## Report of Analysis

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Client Sample ID: OSMW-6D  
 Lab Sample ID: FA41854-2  
 Matrix: AQ - Ground Water  
 Method: SW846 8260C  
 Project: BMSMC, Humacao, PR

Date Sampled: 03/06/17  
 Date Received: 03/08/17  
 Percent Solids: n/a

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	I46111.D	1	03/09/17	WV	n/a	n/a	VI1288
Run #2							

Run #	Purge Volume
Run #1	5.0 ml
Run #2	

CAS No.	Compound	Result	RL	MDL	Units	Q
71-43-2	Benzene	ND	1.0	0.31	ug/l	
67-66-3	Chloroform	ND	1.0	0.30	ug/l	
75-71-8	Dichlorodifluoromethane	ND	2.0	0.50	ug/l	
107-06-2	1,2-Dichloroethane	ND	1.0	0.31	ug/l	
1634-04-4	Methyl Tert Butyl Ether	ND	1.0	0.23	ug/l	
75-85-4	Tert-Amyl Alcohol	ND	20	5.3	ug/l	
75-01-4	Vinyl Chloride	ND	1.0	0.41	ug/l	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
1868-53-7	Dibromofluoromethane	99%		83-118%
17060-07-0	1,2-Dichloroethane-D4	103%		79-125%
2037-26-5	Toluene-D8	103%		85-112%
460-00-4	4-Bromofluorobenzene	101%		83-118%



ND = Not detected      MDL = Method Detection Limit  
 RL = Reporting Limit  
 E = Indicates value exceeds calibration range

J = Indicates an estimated value  
 B = Indicates analyte found in associated method blank  
 N = Indicates presumptive evidence of a compound



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## Report of Analysis

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Client Sample ID:	OSMW-6D	Date Sampled:	03/06/17
Lab Sample ID:	FA41854-2	Date Received:	03/08/17
Matrix:	AQ - Ground Water	Percent Solids:	n/a
Method:	SW846 8270D SW846 3510C		
Project:	BMSMC, Humacao, PR		

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	X052883.D	1	03/14/17	NG	03/11/17	OP64132	SX2241
Run #2							

Run #	Initial Volume	Final Volume
Run #1	1050 ml	1.0 ml
Run #2		

CAS No.	Compound	Result	RL	MDL	Units	Q
100-52-7	Benzaldehyde	ND	24	4.8	ug/l	
117-81-7	bis(2-Ethylhexyl)phthalate <sup>a</sup>	ND	4.8	0.95	ug/l	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
4165-60-0	Nitrobenzene-d5	76%		42-108%
321-60-8	2-Fluorobiphenyl	69%		40-106%
1718-51-0	Terphenyl-d14	71%		39-121%

(a) Associated BS recovery outside control limits.



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 RL = Reporting Limit  
 E = Indicates value exceeds calibration range

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 B = Indicates analyte found in associated method blank  
 N = Indicates presumptive evidence of a compound

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## Report of Analysis

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Client Sample ID:	OSMW-6D	Date Sampled:	03/06/17
Lab Sample ID:	FA41854-2	Date Received:	03/08/17
Matrix:	AQ - Ground Water	Percent Solids:	n/a
Method:	SW846 8270D BY SIM SW846 3510C		
Project:	BMSMC, Humacao, PR		

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	W098089.D	1	03/17/17	FS	03/11/17	OP64133	SW4359
Run #2	U060373.D	1	03/14/17	NJ	03/11/17	OP64133	SU2649

Run #	Initial Volume	Final Volume
Run #1	1050 ml	1.0 ml
Run #2	1050 ml	1.0 ml

CAS No.	Compound	Result	RL	MDL	Units	Q
56-55-3	Benzo(a)anthracene	ND	0.19	0.038	ug/l	
123-91-1	1,4-Dioxane	1.8 <sup>a</sup>	0.29	0.14	ug/l	
91-20-3	Naphthalene	ND	0.95	0.38	ug/l	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
4165-60-0	Nitrobenzene-d5	60% <sup>b</sup>	68% <sup>b</sup>	42-108%
321-60-8	2-Fluorobiphenyl	59% <sup>b</sup>	59% <sup>b</sup>	40-106%
1718-51-0	Terphenyl-d14	52% <sup>b</sup>	64% <sup>b</sup>	39-121%

(a) Result is from Run# 2

(b) Surrogate recoveries corrected for actual spike amount.



ND = Not detected MDL = Method Detection Limit  
 RL = Reporting Limit  
 E = Indicates value exceeds calibration range

J = Indicates an estimated value  
 B = Indicates analyte found in associated method blank  
 N = Indicates presumptive evidence of a compound

SGS Accutest

## Report of Analysis

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Client Sample ID: OSMW-6D  
 Lab Sample ID: FA41854-2  
 Matrix: AQ - Ground Water  
 Method: MADEP VPH REV 1.1  
 Project: BMSMC, Humacao, PR

Date Sampled: 03/06/17  
 Date Received: 03/08/17  
 Percent Solids: n/a

	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	UU019382.D	1	03/15/17	AJC	n/a	n/a	GUU1017
Run #2 <sup>a</sup>	UU019426.D	1	03/17/17	AJC	n/a	n/a	GUU1019

	Purge Volume
Run #1	5.0 ml
Run #2	5.0 ml

## MADEP VPH List

CAS No.	Compound	Result	RL	MDL	Units	Q
	C9- C10 Aromatics (Unadj.)	ND	100	35	ug/l	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
460-00-4	BFB	113%	111%	70-130%
460-00-4	BFB	108%	107%	70-130%

(a) Confirmation run.



ND = Not detected MDL = Method Detection Limit  
 RL = Reporting Limit  
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J = Indicates an estimated value  
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 N = Indicates presumptive evidence of a compound

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## Report of Analysis

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Client Sample ID:	OSMW-6D	Date Sampled:	03/06/17
Lab Sample ID:	FA41854-2	Date Received:	03/08/17
Matrix:	AQ - Ground Water	Percent Solids:	n/a
Method:	MADEP EPH REV 1.1 SW846 3510C		
Project:	BMSMC; Humacao, PR		

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	NN017921.D	1	03/22/17 20:53	MG	03/17/17 17:40	OP64226	GNN902
Run #2							

Run #	Initial Volume	Final Volume
Run #1	1000 ml	2.0 ml
Run #2		

## MAEPH List

CAS No.	Compound	Result	RL	MDL	Units	Q
	C11-C22 Aromatics (Unadj.)	ND	200	80	ug/l	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
3386-33-2	1-Chlorooctadecane	55%		40-140%
580-13-2	2-Bromonaphthalene	98%		40-140%
84-15-1	o-Terphenyl	80%		40-140%
321-60-8	2-Fluorobiphenyl	99%		40-140%



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 N = Indicates presumptive evidence of a compound

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## Report of Analysis

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Client Sample ID: OSMW-6D  
 Lab Sample ID: FA41854-2  
 Matrix: AQ - Ground Water  
 Method: SW846 8081B SW846 3510C  
 Project: BSMC, Humacao, PR

Date Sampled: 03/06/17  
 Date Received: 03/08/17  
 Percent Solids: n/a

	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1 <sup>a</sup>	KK82216.D	1	03/17/17	MV	03/10/17	OP64131	GKK2635
Run #2							

	Initial Volume	Final Volume
Run #1	1000 ml	5.0 ml
Run #2		

CAS No.	Compound	Result	RL	MDL	Units	Q
60-57-1	Dieldrin	ND	0.010	0.0024	ug/l	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
877-09-8	Tetrachloro-m-xylene	107%		42-127%
2051-24-3	Decachlorobiphenyl	57%		27-127%

(a) Associated MS/MSD outside of control limits.



ND = Not detected MDL = Method Detection Limit  
 RL = Reporting Limit  
 E = Indicates value exceeds calibration range

J = Indicates an estimated value  
 B = Indicates analyte found in associated method blank  
 N = Indicates presumptive evidence of a compound

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## Report of Analysis

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Client Sample ID:	TB030617B	Date Sampled:	03/06/17
Lab Sample ID:	FA41854-3	Date Received:	03/08/17
Matrix:	AQ - Trip Blank Water	Percent Solids:	n/a
Method:	SW846 8260C		
Project:	BMSMC, Humacao, PR		

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	I46112.D	1	03/09/17	WV	n/a	n/a	VI1288
Run #2							

Run #	Purge Volume
Run #1	5.0 ml
Run #2	

CAS No.	Compound	Result	RL	MDL	Units	Q
71-43-2	Benzene	ND	1.0	0.31	ug/l	
67-66-3	Chloroform	ND	1.0	0.30	ug/l	
75-71-8	Dichlorodifluoromethane	ND	2.0	0.50	ug/l	
107-06-2	1,2-Dichloroethane	ND	1.0	0.31	ug/l	
1634-04-4	Methyl Tert Butyl Ether	ND	1.0	0.23	ug/l	
75-85-4	Tert-Amyl Alcohol	ND	20	5.3	ug/l	
75-01-4	Vinyl Chloride	ND	1.0	0.41	ug/l	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
1868-53-7	Dibromofluoromethane	99%		83-118%
17060-07-0	1,2-Dichloroethane-D4	102%		79-125%
2037-26-5	Toluene-D8	104%		85-112%
460-00-4	4-Bromofluorobenzene	100%		83-118%



ND = Not detected      MDL = Method Detection Limit  
 RL = Reporting Limit  
 E = Indicates value exceeds calibration range

J = Indicates an estimated value  
 B = Indicates analyte found in associated method blank  
 N = Indicates presumptive evidence of a compound

## Matrix Spike/Matrix Spike Duplicate Summary

Page 1 of 1

Job Number: FA41854

Account: AMANYWP Anderson, Mulholland &amp; Associates

Project: BSMC, Humacao, PR

Sample	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
FA41854-2MS	I46121.D	1	03/09/17	WV	n/a	n/a	VI1288
FA41854-2MSD	I46122.D	1	03/09/17	WV	n/a	n/a	VI1288
FA41854-2	I46111.D	1	03/09/17	WV	n/a	n/a	VI1288

The QC reported here applies to the following samples:

Method: SW846 8260C

FA41854-1, FA41854-2, FA41854-3

CAS No.	Compound	FA41854-2 ug/l	Spike Q	MS ug/l	MS %	Spike ug/l	MSD ug/l	MSD %	RPD	Limits Rec/RPD
71-43-2	Benzene	ND	25	28.9	116	25	27.1	108	6	81-122/14
67-66-3	Chloroform	ND	25	25.4	102	25	24.0	96	6	80-124/15
75-71-8	Dichlorodifluoromethane	ND	25	19.4	78	25	18.7	75	4	42-167/19
107-06-2	1,2-Dichloroethane	ND	25	25.0	100	25	23.6	94	6	75-125/14
1634-04-4	Methyl Tert Butyl Ether	ND	25	23.0	92	25	21.7	87	6	72-117/14
75-85-4	Tert-Amyl Alcohol	ND	250	224	90	250	219	88	2	65-124/23
75-01-4	Vinyl Chloride	ND	25	23.4	94	25	22.0	88	6	69-159/18

CAS No.	Surrogate Recoveries	MS	MSD	FA41854-2	Limits
1868-53-7	Dibromofluoromethane	103%	100%	99%	83-118%
17060-07-0	1,2-Dichloroethane-D4	106%	104%	103%	79-125%
2037-26-5	Toluene-D8	99%	98%	103%	85-112%
460-00-4	4-Bromofluorobenzene	97%	103%	101%	83-118%



\* = Outside of Control Limits.

## Matrix Spike/Matrix Spike Duplicate Summary

Page 1 of 1

Job Number: FA41854

Account: AMANYWP Anderson, Mulholland &amp; Associates

Project: BSMC, Humacao, PR

Sample	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
OP64132-MS	X052884.D	1	03/14/17	NG	03/11/17	OP64132	SX2241
OP64132-MSD	X052885.D	1	03/14/17	NG	03/11/17	OP64132	SX2241
FA41854-2	X052883.D	1	03/14/17	NG	03/11/17	OP64132	SX2241

The QC reported here applies to the following samples:

Method: SW846 8270D

FA41854-1, FA41854-2

CAS No.	Compound	FA41854-2 ug/l	Spike Q	MS ug/l	MS %	Spike ug/l	MSD ug/l	MSD %	RPD	Limits Rec/RPD
100-52-7	Benzaldehyde	ND	96.2	91.1	95	96.2	91.2	95	0	36-129/29
117-81-7	bis(2-Ethylhexyl)phthalate	ND	96.2	142	148*	96.2	133	138*	7	61-117/23

CAS No.	Surrogate Recoveries	MS	MSD	FA41854-2	Limits
367-12-4	2-Fluorophenol	77%* a	79%* a		14-67%
4165-62-2	Phenol-d5	91%* a	89%* a		10-50%
118-79-6	2,4,6-Tribromophenol	102%	100%		33-118%
4165-60-0	Nitrobenzene-d5	103%	105%	76%	42-108%
321-60-8	2-Fluorobiphenyl	98%	98%	69%	40-106%
1718-51-0	Terphenyl-d14	102%	97%	71%	39-121%

(a) Outside control limits.



\* = Outside of Control Limits.



## Matrix Spike/Matrix Spike Duplicate Summary

Page 1 of 1

Job Number: FA41854

Account: AMANYWP Anderson, Mulholland &amp; Associates

Project: BSMC, Humacao, PR

Sample	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
OP64133-MS	U060374.D	1	03/14/17	NJ	03/11/17	OP64133	SU2649
OP64133-MSD	U060375.D	1	03/14/17	NJ	03/11/17	OP64133	SU2649
FA41854-2	U060373.D	1	03/14/17	NJ	03/11/17	OP64133	SU2649

The QC reported here applies to the following samples:

Method: SW846 8270D BY SIM

FA41854-1, FA41854-2

CAS No.	Compound	FA41854-2 ug/l	Spike Q	MS ug/l	MS %	Spike ug/l	MSD ug/l	MSD %	RPD	Limits Rec/RPD
123-91-1	1,4-Dioxane	1.8	19.2	5.0	17	19.2	5.1	17	2	15-69/31

CAS No.	Surrogate Recoveries	MS	MSD	FA41854-2	Limits
4165-60-0	Nitrobenzene-d5	70% <sup>a</sup>	74% <sup>a</sup>	68% <sup>a</sup>	42-108%
321-60-8	2-Fluorobiphenyl	68% <sup>a</sup>	70% <sup>a</sup>	59% <sup>a</sup>	40-106%
1718-51-0	Terphenyl-d14	76% <sup>a</sup>	72% <sup>a</sup>	64% <sup>a</sup>	39-121%

(a) Surrogate recoveries corrected for actual spike amount.



\* = Outside of Control Limits.

## Matrix Spike/Matrix Spike Duplicate Summary

Page 1 of 1

Job Number: FA41854

Account: AMANYWP Anderson, Mulholland &amp; Associates

Project: BMSMC, Humacao, PR

Sample	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
OP64133-MS	W098090.D	1	03/17/17	FS	03/11/17	OP64133	SW4359
OP64133-MSD	W098091.D	1	03/17/17	FS	03/11/17	OP64133	SW4359
FA41854-2	W098089.D	1	03/17/17	FS	03/11/17	OP64133	SW4359

The QC reported here applies to the following samples:

Method: SW846 8270D BY SIM

FA41854-1, FA41854-2

CAS No.	Compound	FA41854-2 ug/l	Spike Q	MS ug/l	MS %	Spike ug/l	MSD ug/l	MSD %	RPD	Limits Rec/RPD
56-55-3	Benzo(a)anthracene	ND	9.62	9.3	97	9.62	9.2	96	1	65-106/22
91-20-3	Naphthalene	ND	19.2	16.4	85	19.2	16.1	84	2	56-105/27

CAS No.	Surrogate Recoveries	MS	MSD	FA41854-2	Limits
118-79-6	2,4,6-Tribromophenol	113%	108%		33-118%
4165-60-0	Nitrobenzene-d5	101%	97%	60% <sup>a</sup>	42-108%
321-60-8	2-Fluorobiphenyl	97%	103%	59% <sup>a</sup>	40-106%
1718-51-0	Terphenyl-d14	82%	82%	52% <sup>a</sup>	39-121%

(a) Surrogate recoveries corrected for actual spike amount.



\* = Outside of Control Limits.

## Matrix Spike/Matrix Spike Duplicate Summary

Page 1 of 1

Job Number: FA41854

Account: AMANYWP Anderson, Mulholland &amp; Associates

Project: BMSMC, Humacao, PR

Sample	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
OP64131-MS	KK82217.D	1	03/17/17	MV	03/10/17	OP64131	GKK2635
OP64131-MSD	KK82218.D	1	03/17/17	MV	03/10/17	OP64131	GKK2635
FA41854-2 <sup>a</sup>	KK82216.D	1	03/17/17	MV	03/10/17	OP64131	GKK2635

The QC reported here applies to the following samples:

Method: SW846 8081B

FA41854-1, FA41854-2

CAS No.	Compound	FA41854-2 ug/l	Spike Q	MS ug/l	MS %	Spike ug/l	MSD ug/l	MSD %	RPD	Limits Rec/RPD
60-57-1	Dieldrin	ND	0.5	0.59	118	0.5	0.62	124	5	66-138/22

CAS No.	Surrogate Recoveries	MS	MSD	FA41854-2	Limits
877-09-8	Tetrachloro-m-xylene	114%	118%	107%	42-127%
2051-24-3	Decachlorobiphenyl	118%	110%	57%	27-127%

(a) Associated MS/MSD outside of control limits.

12.4.3  
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\* = Outside of Control Limits.



FL

## CHAIN OF CUSTODY

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PAGE 1 OF 1

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**FA41854: Chain of Custody**  
**Page 1 of 3**

## EXECUTIVE NARRATIVE

SDG No: FA41854 Laboratory: Accutest, Florida  
Analysis: SW846-8260C Number of Samples: 5  
Location: BMSMC – Humacao, PR

**SUMMARY:** Five (5) samples were analyzed for selected volatile organic compounds (VOA Special List) by method SW846-8260C. The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: USEPA Hazardous Waste Support Section SOP No. HW-33A Revision 0 SOM02.2. Low/Medium Volatile Data Validation. July, 2015. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

**Critical issues:** None  
**Major:** None  
**Minor:** None

**Critical findings:** None  
**Major findings:** None  
**Minor findings:** None

**COMMENTS:** Results are valid and can be used for decision making purposes.

**Reviewers Name:** Rafael Infante  
Chemist License 1888

**Signature:**

**Date:**

  
April 14, 2017

# ORGANIC DATA SAMPLE SUMMARY

Sample ID: FA41854-1

Sample location: BMSMC, Humacao, PR

Sampling date: 3/6/2017

Matrix: Groundwater

## METHOD: 8260C

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Benzene	1.0	ug/L	1.0	-	U	Yes
Chloroform	1.0	ug/L	1.0	-	U	Yes
Dichlorodifluoromethane	2.0	ug/L	1.0	-	U	Yes
1,2-Dichloroethane	1.0	ug/L	1.0	-	U	Yes
Methyl Tert Butyl Ether	1.0	ug/L	1.0	-	U	Yes
Tert-Amyl Alcohol	20	ug/L	1.0	-	U	Yes
Vinyl chloride	1.0	ug/L	1.0	-	U	Yes

Sample ID: FA41854-2

Sample location: BMSMC, Humacao, PR

Sampling date: 3/6/2017

Matrix: Groundwater

## METHOD: 8260C

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Benzene	1.0	ug/L	1.0	-	U	Yes
Chloroform	1.0	ug/L	1.0	-	U	Yes
Dichlorodifluoromethane	2.0	ug/L	1.0	-	U	Yes
1,2-Dichloroethane	1.0	ug/L	1.0	-	U	Yes
Methyl Tert Butyl Ether	1.0	ug/L	1.0	-	U	Yes
Tert-Amyl Alcohol	20	ug/L	1.0	-	U	Yes
Vinyl chloride	1.0	ug/L	1.0	-	U	Yes

Sample ID: FA41854-3

Sample location: BMSMC, Humacao, PR

Sampling date: 3/6/2017

Matrix: AQ - Trip Blank Water

METHOD: 8260C

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Benzene	1.0	ug/L	1.0	-	U	Yes
Chloroform	1.0	ug/L	1.0	-	U	Yes
Dichlorodifluoromethane	2.0	ug/L	1.0	-	U	Yes
1,2-Dichloroethane	1.0	ug/L	1.0	-	U	Yes
Methyl Tert Butyl Ether	1.0	ug/L	1.0	-	U	Yes
Tert-Amyl Alcohol	20	ug/L	1.0	-	U	Yes
Vinyl chloride	1.0	ug/L	1.0	-	U	Yes

Sample ID: FA41854-2MS

Sample location: BMSMC, Humacao, PR

Sampling date: 3/6/2017

Matrix: Groundwater

METHOD: 8260C

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Benzene	28.9	ug/L	1.0	-	-	Yes
Chloroform	25.4	ug/L	1.0	-	-	Yes
Dichlorodifluoromethane	19.4	ug/L	1.0	-	-	Yes
1,2-Dichloroethane	25.0	ug/L	1.0	-	-	Yes
Methyl Tert Butyl Ether	23.0	ug/L	1.0	-	-	Yes
Tert-Amyl Alcohol	224	ug/L	1.0	-	-	Yes
Vinyl chloride	25.0	ug/L	1.0	-	-	Yes

Sample ID: FA41854-2MSD

Sample location: BMSMC, Humacao, PR

Sampling date: 3/6/2017

Matrix: Groundwater

METHOD: 8260C

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Benzene	27.1	ug/L	1.0	-	-	Yes
Chloroform	24.0	ug/L	1.0	-	-	Yes
Dichlorodifluoromethane	18.7	ug/L	1.0	-	-	Yes
1,2-Dichloroethane	23.6	ug/L	1.0	-	-	Yes
Methyl Tert Butyl Ether	21.7	ug/L	1.0	-	-	Yes
Tert-Amyl Alcohol	219	ug/L	1.0	-	-	Yes
Vinyl chloride	22.0	ug/L	1.0	-	-	Yes



# DATA REVIEW WORKSHEETS

Project Number: FA41854  
 Date: March 6, 2017  
 Shipping date: March 7, 2017  
 EPA Region: 2

## REVIEW OF VOLATILE ORGANIC PACKAGE Low/Medium Volatile Data Validation

The following guidelines for evaluating volatile organics were created to delineate required validation actions. This document will assist the reviewer in using professional judgment to make more informed decision and in better serving the needs of the data users. The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: USEPA Hazardous Waste Support Section SOP No. HW-33A Revision 0 SOM02.2. Low/Medium Volatile Data Validation. July, 2015. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

The hardcopied (laboratory name) Accutest - Orlando data package received has been reviewed and the quality control and performance data summarized. The data review for VOCs included:

Lab. Project/SDG No.: FA41854 Sample matrix: Groundwater  
 No. of Samples: 5  
 Trip blank No.: FA41854-3  
 Field blank No.: -  
 Equipment blank No.: -  
 Field duplicate No.: -

<input checked="" type="checkbox"/> Data Completeness	<input checked="" type="checkbox"/> Laboratory Control Spikes
<input checked="" type="checkbox"/> Holding Times	<input checked="" type="checkbox"/> Field Duplicates
<input checked="" type="checkbox"/> GC/MS Tuning	<input checked="" type="checkbox"/> Calibrations
<input checked="" type="checkbox"/> Internal Standard Performance	<input checked="" type="checkbox"/> Compound Identifications
<input checked="" type="checkbox"/> Blanks	<input checked="" type="checkbox"/> Compound Quantitation
<input checked="" type="checkbox"/> Surrogate Recoveries	<input checked="" type="checkbox"/> Quantitation Limits
<input checked="" type="checkbox"/> Matrix Spike/Matrix Spike Duplicate	

Overall Comments: Selected\_VOA\_from\_the\_special\_ist\_(SW846\_8260C).

### Definition of Qualifiers:

J- Estimated results  
 U- Compound not detected  
 R- Rejected data  
 UJ- Estimated nondetect

Reviewer: Rafael Defaut  
 Date: April 14, 2017

100

DATE RECEIVED

This image shows a single sheet of white paper with horizontal blue or grey ruling lines. A prominent dashed diagonal line runs across the page from the upper-left corner towards the lower-right corner. The lines are evenly spaced and extend across the width of the page. There is no handwriting or other markings on the paper.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met  
and/or see below       

### HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE ANALYZED	pH	ACTION
All samples analyzed within method recommended holding. Samples properly preserved.				

### Criteria

Aqueous samples – 14 days from sample collection for preserved samples ( $\text{pH} \leq 2$ ,  $4 \pm 2^\circ\text{C}$ ), no air bubbles.

Aqueous samples – 7 days from sample collection for unpreserved samples,  $4^\circ\text{C}$ , no air bubbles.

Soil samples- 14 days from sample collection.

Cooler temperature (Criteria:  $4 \pm 2^\circ\text{C}$ ):  $3.0/3.2^\circ\text{C}$  - OK

### Actions

#### **Aqueous samples**

- If there is no evidence that the samples were properly preserved ( $\text{pH} < 2$ ,  $T = 4^\circ\text{C} \pm 2^\circ\text{C}$ ), but the samples were analyzed within the technical holding time [7 days from sample collection], no qualification of the data is necessary.
- If there is no evidence that the samples were properly preserved, and the samples were analyzed outside of the technical holding time [7 days from sample collection], qualify detects for all volatile compounds as estimated (J) and non-detects as unusable (R).
- If the samples were properly preserved, and the samples were analyzed within the technical holding time [14 days from sample collection], no qualification of the data is necessary.
- If the samples were properly preserved, but were analyzed outside of the technical holding time [14 days from sample collection], qualify detects as estimated (J) and non-detects as unusable (R).
- If air bubbles were present in the sample vial used for analysis, qualify detected compounds as estimated (J-) and non-detected compounds as estimated (UJ).

#### **Non-aqueous samples**

- If there is no evidence that the samples were properly preserved ( $T < -7^\circ\text{C}$  or  $T = 4^\circ\text{C} \pm 2^\circ\text{C}$  and preserved with  $\text{NaHSO}_4$ ), but the samples were analyzed within the technical holding time [14 days

## DATA REVIEW WORKSHEETS

from sample collection], qualify detects for all volatile compounds as estimated (J) and non-detects as (UJ) or unusable (R) using professional judgment.

b. If the samples were properly preserved, and the samples were analyzed within the technical holding time [14 days from sample collection], no qualification of the data is necessary.

c. If there is no evidence that the samples were properly preserved, and the samples were analyzed outside of the technical holding time [14 days from sample collection], qualify detects for all volatile compounds as estimated (J) and non-detects as unusable (R).

d. If the samples were properly preserved, but were analyzed outside of the technical holding time [14 days from sample collection], qualify detects as estimated (J) and non-detects as unusable (R).

### **Qualify TCLP/SPLP samples**

a. If the TCLP/SPLP ZHE procedure is performed within the extraction technical holding time of 14 days, detects and non-detects should not be qualified.

b. If the TCLP/SPLP ZHE procedure is performed outside the extraction technical holding time of 14 days, qualify detects as estimated (J) and non-detects as unusable (R).

c. If TCLP/SPLP aqueous samples and TCLP/SPLP leachate samples are analyzed within the technical holding time of 7 days, detects and non-detects should not be qualified.

d. If TCLP/SPLP aqueous samples and TCLP/SPLP leachate samples are analyzed outside of the technical holding time of 7 days, qualify detects as estimated (J) and non-detects as unusable (R).

DATA REVIEW WORKSHEETS

Table 1. Holding Time Actions for Low/Medium Volatile Analyses - Summary

Matrix	Preserved	Criteria	Action	
			Detected Associated Compounds	Non-Detected Associated Compounds
Aqueous	No	≤ 7 days	No qualification	
	No	> 7 days	J	R
	Yes	≤ 14 days	No qualification	
	Yes	> 14 days	J	R
Non-Aqueous	No	≤ 14 days	J	Professional judgment. UJ or R
	Yes	≤ 14 days	No qualification	
	Yes No	> 14 days	J	R
TCLP SPLP	Yes	≤ 14 days	No qualification	
TCLP SPLP	No	> 14 days	J	R
TCLP SPLP	ZHE performed within the 14-day technical holding time		No qualification	
TCLP SPLP	ZHE performed outside the 14-day technical holding time		J	R
TCLP SPLP aqueous & TCLP SPLP leachate	Analyzed within 7 days		No qualification	
TCLP SPLP aqueous & TCLP SPLP leachate	Analyzed outside 7 days		J	R
Sample temperature outside 4°C ± 2°C upon receipt at the laboratory			Use professional judgment	
Holding times grossly exceeded			J	R

## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met see below       

### GC/MS TUNING

The assessment of the tuning results is to determine if the sample instrumentation is within the standard tuning QC limits

  X   The BFB performance results were reviewed and found to be within the specified criteria.

  X   BFB tuning was performed for every 12 hours of sample analysis.

**NOTES:** All mass spectrometer instrument conditions must be identical to those used during the sample analysis. Background subtraction actions resulting in spectral distortions for the sole purpose of meeting the method specifications are contrary to the Quality Assurance (QA) objectives, and are therefore unacceptable.

**NOTES:** No data should be qualified based on BFB failure. Instances of this should be noted in the narrative.

All ion abundance ratios must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120% that of m/z 95.

#### Actions:

If samples are analyzed without a preceding valid instrument performance check, qualify all data in those samples as unusable (R).

If ion abundance criteria are not met, professional judgment may be applied to determine to what extent the data may be utilized. When applying professional judgment to this topic, the most important factors to consider are the empirical results that are relatively insensitive to location on the chromatographic profile and the type of instrumentation. Therefore, the critical ion abundance criteria for BFB are the m/z 95/96, 174/175, 174/176, and 176/177 ratios. The relative abundances of m/z 50 and 75 are of lower importance. This issue is more critical for Tentatively Identified Compounds (TICs) than for target analytes.

**Note:** State in the Data Review Narrative, decisions to use analytical data associated with BFB instrument performance checks not meeting contract requirements.

**Note:** Verify that that instrument instrument performance check criteria were achieved using techniques described in Low/Medium Volatiles Organic Analysis, Section II.D.5 of the SOM02.2 NFG, obtain additional information on the instrument performance checks. Make sure that background subtraction was performed from the BFB peak and not from background subtracting from the solvent front or from another region of the chromatogram.

## DATA REVIEW WORKSHEETS

Use professional judgment to determine whether associated data should be qualified based on the spectrum of the mass calibration compound.

List the samples affected:

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If mass calibration is in error, all associated data are rejected.

## DATA REVIEW WORKSHEETS

All criteria were met X  
 Criteria were not met  
 and/or see below \_\_\_\_\_

### CALIBRATION VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration: 02/28/17  
 Dates of continuing (initial) calibration: 02/28/17  
 Dates of continuing calibration: 03/09/17  
 Dates of ending calibration: 02/28/17; 03/09/17  
 Instrument ID numbers: GCMSI  
 Matrix/Level: Aqueous/low

DATE	LAB ID#	FILE	CRITERIA OUT RFs, %RSD, %D, r	COMPOUND	SAMPLES AFFECTED

**Note:** Initial calibration, initial calibration verification, and continuing calibration verification within the method and validation guidance document required performance criteria. Closing calibration check verification included in data package.

#### Criteria

The analyte calibration criteria in the following Table must be obtained. Analytes not meeting the criteria are qualified.

A separate worksheet should be filled for each initial curve.



DATA REVIEW WORKSHEETS

Initial Calibration - Table 2. RRF, %RSD, and %D Acceptance Criteria for Initial Calibration and CCV for Low/Medium Volatile Analysis

Analyte	Minimum RRF	Maximum %RSD	Opening Maximum %D <sup>1</sup>	Closing Maximum %D
Dichlorodifluoromethane	0.010	25.0	±40.0	±50.0
Chloromethane	0.010	20.0	±30.0	±50.0
Vinyl chloride	0.010	20.0	±25.0	±50.0
Bromomethane	0.010	40.0	±30.0	±50.0
Chloroethane	0.010	40.0	±25.0	±50.0
Trichlorofluoromethane	0.010	40.0	±30.0	±50.0
1,1-Dichloroethene	0.060	20.0	±20.0	±25.0
1,1,2-Trichloro-1,2,2-trifluoroethane	0.050	25.0	±25.0	±50.0
Acetone	0.010	40.0	±40.0	±50.0
Carbon disulfide	0.100	20.0	±25.0	±25.0
Methyl acetate	0.010	40.0	±40.0	±50.0
Methylene chloride	0.010	40.0	±30.0	±50.0
trans-1,2-Dichloroethene	0.100	20.0	±20.0	±25.0
Methyl tert-butyl ether	0.100	40.0	±25.0	±50.0
1,1-Dichloroethane	0.300	20.0	±20.0	±25.0
cis-1,2-Dichloroethene	0.200	20.0	±20.0	±25.0
2-Butanone	0.010	40.0	±40.0	±50.0
Bromochloromethane	0.100	20.0	±20.0	±25.0
Chloroform	0.300	20.0	±20.0	±25.0
1,1,1-Trichloroethane	0.050	20.0	±25.0	±25.0
Cyclohexane	0.010	40.0	±25.0	±50.0
Carbon tetrachloride	0.100	20.0	±25.0	±25.0
Benzene	0.200	20.0	±20.0	±25.0
1,2-Dichloroethane	0.070	20.0	±20.0	±25.0
Trichloroethene	0.200	20.0	±20.0	±25.0
Methylcyclohexane	0.050	40.0	±25.0	±50.0
1,2-Dichloropropane	0.200	20.0	±20.0	±25.0
Bromodichloromethane	0.300	20.0	±20.0	±25.0
cis-1,3-Dichloropropene	0.300	20.0	±20.0	±25.0
4-Methyl-2-pentanone	0.030	25.0	±30.0	±50.0
Toluene	0.300	20.0	±20.0	±25.0
trans-1,3-Dichloropropene	0.200	20.0	±20.0	±25.0
1,1,2-Trichloroethane	0.200	20.0	±20.0	±25.0
Tetrachloroethene	0.100	20.0	±20.0	±25.0
2-Hexanone	0.010	40.0	±40.0	±50.0
Dibromochloromethane	0.200	20.0	±20.0	±25.0
1,2-Dibromoethane	0.200	20.0	±20.0	±25.0
Chlorobenzene	0.400	20.0	±20.0	±25.0
Ethylbenzene	0.400	20.0	±20.0	±25.0

# DATA REVIEW WORKSHEETS

Analyte	Minimum RRF	Maximum %RSD	Opening Maximum %D <sup>1</sup>	Closing Maximum
m,p-Xylene	0.200	20.0	±20.0	±25.0
o-Xylene	0.200	20.0	±20.0	±25.0
Styrene	0.200	20.0	±20.0	±25.0
Bromoform	0.100	20.0	±25.0	±50.0
Isopropylbenzene	0.400	20.0	±25.0	±25.0
1,1,2,2-Tetrachloroethane	0.200	20.0	±25.0	±25.0
1,3-Dichlorobenzene	0.500	20.0	±20.0	±25.0
1,4-Dichlorobenzene	0.600	20.0	±20.0	±25.0
1,2-Dichlorobenzene	0.600	20.0	±20.0	±25.0
1,2-Dibromo-3-chloropropane	0.010	25.0	±30.0	±50.0
1,2,4-Trichlorobenzene	0.400	20.0	±30.0	±50.0
1,2,3-Trichlorobenzene	0.400	25.0	±30.0	±50.0
<b>Deuterated Monitoring Compound</b>				
Vinyl chloride-d <sub>3</sub>	0.010	20.0	±30.0	±50.0
Chloroethane-d <sub>3</sub>	0.010	40.0	±30.0	±50.0
1,1-Dichloroethene-d <sub>2</sub>	0.050	20.0	±25.0	±25.0
2-Butanone-d <sub>6</sub>	0.010	40.0	±40.0	±50.0
Chloroform-d	0.300	20.0	±20.0	±25.0
1,2-Dichloroethane-d <sub>4</sub>	0.060	20.0	±25.0	±25.0
Benzene-d <sub>6</sub>	0.300	20.0	±20.0	±25.0
1,2-Dichloropropane-d <sub>6</sub>	0.200	20.0	±20.0	±25.0
Toluene-d <sub>8</sub>	0.300	20.0	±20.0	±25.0
trans-1,3-Dichloropropene-d <sub>4</sub>	0.200	20.0	±20.0	±25.0
2-Hexanone-d <sub>8</sub>	0.010	40.0	±40.0	±50.0
1,1,2,2-Tetrachloroethane-d <sub>2</sub>	0.200	20.0	±25.0	±25.0
1,2-Dichlorobenzene-d <sub>4</sub>	0.400	20.0	±20.0	±25.0

- <sup>1</sup> If a closing CCV is acting as an opening CCV, all target analytes and DMCs must meet the requirements for an opening CCV.

## Actions:

1. If any volatile target compound has an RRF value less than the minimum in the table, use professional judgment for detects, based on mass spectral identification, to qualify the data as estimated (J+ or R).
  - a. If any volatile target compound has an RRF value less than the minimum criterion, qualify non-detected compounds as unusable (R).
  - b. If any of the volatile target compounds listed in the Table has %RSD greater than the criteria, qualify detects as estimated (J), and non-detected compounds using professional judgment.
  - c. If the volatile target compounds meet the acceptance criteria for RRF and the %RSD, no qualification of the data is necessary.

## DATA REVIEW WORKSHEETS

- d. No qualification of the data is necessary on the DMC RRF and %RSD data alone. Use professional judgment and follow the guidelines in Action 2 to evaluate the DMC RRF and %RSD data in conjunction with the DMC recoveries to determine the need for qualification of data.
2. At the reviewer's discretion, and based on the project-specific Data Quality Objectives (DQOs), a more in-depth review may be considered using the following guidelines:
    - a. If any volatile target compound has a %RSD greater than the maximum criterion in the Table, and if eliminating either the high or the low-point of the curve does not restore the %RSD to less than or equal to the required maximum:
      - i. Qualify detects for that compound(s) as estimated (J).
      - ii. Qualify non-detected volatile target compounds using professional judgment.
    - b. If the high-point of the curve is outside of the linearity criteria (e.g., due to saturation):
      - i. Qualify detects outside of the linear portion of the curve as estimated (J).
      - ii. No qualifiers are required for detects in the linear portion of the curve.
      - iii. No qualifiers are required for volatile target compounds that were not detected.
    - c. If the low-point of the curve is outside of the linearity criteria:
      - i. Qualify low-level detects in the area of non-linearity as estimated (J).
      - ii. No qualifiers are required for detects in the linear portion of the curve.
      - iii. For non-detected volatile compounds, use the lowest point of the linear portion of the curve to determine the new quantitation limit.

**Note:** If the laboratory has failed to provide adequate calibration information, inform the Region's designated representative to contact the laboratory and request the necessary information. If the information is not available, the reviewer must use professional judgment to assess the data.

State in the Data Review Narrative, if possible, the potential effects on the data due to calibration criteria exceedance.

Note, for the Laboratory COR action, if calibration criteria are grossly exceeded.

Table. Initial Calibration Actions for Low/Medium Volatile Analysis – Summary

Criteria	Action	
	Detect	Non-detect
Initial Calibration not performed at specified frequency and sequence	Use professional judgment R	Use professional judgment R
Initial Calibration not performed at the specified concentrations	J	UJ
RRF < Minimum RRF in Table for target analyte	Use professional judgment J+ or R	R
RRF > Minimum RRF in Table for target analyte	No qualification	No qualification
%RSD > Maximum %RSD in Table for target analyte	J	Use professional judgment
%RSD ≤ Maximum %RSD in Table for target analyte	No qualification	No qualification

All criteria were met   X    
Criteria were not met  
and/or see below           

### Continuing Calibration Verification (CCV)

**NOTE:** Verify that the CCV was run at the required frequency (an opening and closing CCV must be run within 12-hour period) and the CCV was compared to the correct initial calibration. If the mid-point standard from the initial calibration is used as an opening CCV, verify that the result (RRF) of the mid-point standard was compared to the average RRF from the correct initial calibration.

The closing CCV used to bracket the end of a 12-hour analytical sequence may be used as the opening CCV for the new 12-hour analytical sequence, provided that all the technical acceptance criteria are met for an opening CCV (see criteria show before in the Table) . If the closing CCV does not meet the technical acceptance criteria for an opening CCV, then a BFB tune followed by an opening CCV is required and the next 12-hour time period begins with the BFB tune.

All DMCs must meet RRF criteria. No qualification of the data is necessary on the DMCs RRF and %RSD/%D data alone. However, use professional judgment to evaluate the DMC and %RSD/%D data in conjunction with the DMC recoveries to determine the need of qualification the data.

#### Action:

1. If a CCV (opening and closing) was not run at the appropriate frequency, qualify data using professional judgment.
2. Qualify all volatile target compounds in Table shown before using the following criteria:
  - a. For an opening CCV, if any volatile target compound has an RRF value less than the minimum criterion, use professional judgment for detects, based on mass spectral identification, to qualify the data as estimated (J) and qualify non-detected compounds as unusable (R).
  - b. For a closing CCV, if any volatile target compound has an RRF value less than the criteria, use professional judgment for detects based on mass spectral identification to qualify the data as estimated (J), and qualify non-detected compounds as unusable (R).
  - c. For an opening CCV, if the Percent Difference value for any of the volatile target compounds is outside the limits in calibration criteria Table shown before, qualify detects as estimated (J) and non-detected compounds as estimated (UJ).
  - d. For a closing CCV, if the Percent Difference value for any volatile target compound is outside the limits in calibration criteria table, qualify detects as estimated (J) and non-detected compounds as estimated (UJ).
  - e. If the volatile target compounds meet the acceptable criteria for RRF and the Percent Difference, no qualification of the data is necessary.

## DATA REVIEW WORKSHEETS

- f. No qualification of the data is necessary on the DMC RRF and the Percent Difference data alone. Use professional judgment to evaluate the DMC RRF and Percent Difference data in conjunction with the DMC recoveries to determine the need for qualification of data.

Notes: If the laboratory has failed to provide adequate calibration information, inform the Region's designated representative to contact the laboratory and request the necessary information. If the information is not available, the reviewer must use professional judgment to assess the data.

State in the Data Review Narrative, if possible, the potential effects on the data due to calibration criteria exceedance.

Note, for Contract Laboratory COR action, if calibration criteria are grossly exceeded.

Table. Continuing Calibration Actions for Low/Medium Volatile Analysis – Summary

Criteria for Opening CCV	Criteria for Closing CCV	Action	
		Detect	Non-detect
CCV not performed at required frequency	CCV not performed at required frequency	Use professional judgment R	Use professional judgment R
CCV not performed at specified concentration	CCV not performed at specified concentration	Use professional judgment	Use professional judgment
RRF Minimum RRF in Table 2 for target analyte	RRF Minimum RRF in Table for target analyte	Use professional judgment J or R	R
RRF Minimum RRF in Table 2 for target analyte	RRF Minimum RRF in Table for target analyte	No qualification	No qualification
%D outside the Opening Maximum %D limits in Table 2 for target analyte	%D outside the Closing Maximum %D limits in Table for target analyte	J	UJ
%D within the inclusive Opening Maximum %D limits in Table 2 for target analyte	%D within the inclusive Closing Maximum %D limits in Table for target analyte	No qualification	No qualification

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met  
 and/or see below           

### BLANK ANALYSIS RESULTS (Sections 1 & 2)

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

List the contamination in the blanks below. High and low levels blanks must be treated separately.

The concentration of a target analyte in any blank must not exceed its Contract Required Quantitation Limit (CRQL) (2x CRQLs for Methylene chloride, Acetone, and 2-Butanone). TIC concentration in any blanks must be  $\leq 5.0$   $\mu\text{g/L}$  for water (0.0050 mg/L for TCLP leachate) and  $\leq 5.0$   $\mu\text{g/kg}$  for soil matrices.

#### Laboratory blanks

The method blank, like any other sample in the SDG, must meet the technical acceptance criteria for sample analysis.

DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
_No_target_analyte_detected_in_method_blanks._				

#### Field/Equipment/Trip blank

If field or trip blanks are present, the data reviewer should evaluate this data in a similar fashion as the method blanks.

DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
_No_field/equipment_blanks_analyzed_associated_with_this_data_package._No_target_analytes_detected_in_trip_blank.				

**Note:**

## DATA REVIEW WORKSHEETS

All criteria were met ☒   
 Criteria were not met   
 and/or see below \_\_\_\_\_

### BLANK ANALYSIS RESULTS (Section 3)

#### Blank Actions

**Note:** All fields blank results associated with a particular group of samples (may exceed one per case) must be used to qualify data. Trip blanks are used to qualify only those samples with which they were shipped. Blanks may not be qualified because of contamination in another blank. Field blanks and trip blanks must be qualified for system monitoring compounds, instrument performance criteria, and spectral or calibration QC problems.

Samples taken from a drinking water tap do not have associated field blanks.

When applied as described in the Table below, the contaminant concentration in the blank is multiplied by the sample dilution factor.

**Table. Blank and TCLP/SPLP LEB Actions for Low/Medium Volatile Analysis**

Blank Type	Blank Result	Sample Result	Action for Samples
Method. Storage. Field. Trip. TCLP/SPLP LEB. Instrument**	Detects	Not detected	No qualification required
	< CRQL *	< CRQL *	Report CRQL value with a U
		≥ CRQL *	No qualification required
	> CRQL *	< CRQL *	Report CRQL value with a U
		≥ CRQL * and ≤ blank concentration	Report blank value for sample concentration with a U
		≥ CRQL * and > blank concentration	No qualification required
	= CRQL *	≤ CRQL *	Report CRQL value with a U
		> CRQL *	No qualification required
	Gross contamination	Detects	Report blank value for sample concentration with a U

\* 2x the CRQL for methylene chloride, 2-butanone and acetone.

\*\* Qualifications based on instrument blank results affect only the sample analyzed immediately after the sample that has target compounds that exceed the calibration range or non-target compounds that exceed 100 µg/L.

Action Levels (ALs) should be based upon the highest concentration of contaminant determined in any blank. Do not qualify any blank with another blank. The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. No positive sample results should be reported unless the concentration of the compound in the samples exceeds the ALs:

**Notes:**

Compounds qualified "U" for blank contamination are still considered "hits" when qualifying for calibration criteria.

[illegible]



All criteria were met X  
 Criteria were not met  
 and/or see below \_\_\_\_\_

### DEUTERATED MONITORING COMPOUNDS (DMCs)

Laboratory performance of individual samples is established by evaluation of surrogate spike (DMCs) recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

**Table. Volatile Deuterated Monitoring Compounds (DMCs) and Recovery Limits**

DMC	%R for Water Sample	%R for Soil Sample
Vinyl chloride-d3	60-135	30-150
Chloroethane-d5	70-130	30-150
1,1-Dichloroethene-d2	60-125	45-110
2-Butanone-d5	40-130	20-135
Chloroform-d	70-125	40-150
1,2-Dichloroethane-d4	70-125	70-130
Benzene-d6	70-125	20-135
1,2-Dichloropropane-d6	70-120	70-120
Toluene-d8	80-120	30-130
trans-1,3-Dichloropropene-d4	60-125	30-135
2-Hexanone-d5	45-130	20-135
1,1,2,2-Tetrachloroethane-d2	65-120	45-120
1,2-Dichlorobenzene-d4	80-120	75-120

**NOTE:** The recovery limits for any of the compounds listed in the above Table may be expanded at any time during the period of performance if the United States Environmental Protection Agency (EPA) determines that the limits are too restrictive.

#### Action:

Are recoveries for DMCs in volatile samples and blanks must be within the limits specified in the Table above. Yes? or No?

**NOTE:** The recovery limits for any of the compounds listed in the Table above may be expanded at any time during the period of performance if USEPA determines that the limits are too restrictive.

## DATA REVIEW WORKSHEETS

List the DMCs that may fail to meet the recovery limits

Sample ID	Date	DMCs	% Recovery	Action

**Note:** DMCs recoveries within the laboratory required control limits and within the guidance document performance criteria (80 – 120). Other non-deuterated surrogates added to the samples within laboratory control limits.

**Note:** Any sample which has more than 3 DMCs outside the limits must be reanalyzed.

**Action:**

- For any recovery greater than the upper acceptance limit:
  - Qualify detected associated volatile target compounds as estimated high (J+).
  - Do not qualify non-detected associated volatile target compounds.
- For any recovery greater than or equal to 10%, and less than the lower acceptance limit:
  - Qualify detected associated volatile target compounds as estimated low (J-).
  - Qualify non-detected associated volatile target compounds as estimated (UJ).
- For any recovery less than 10%:
  - Qualify detected associated volatile target compounds as estimated low (J-).
  - Qualify non-detected associated volatile target compounds as unusable (R).
- For any recovery within acceptance limits, no qualification of the data is necessary.
- In the special case of a blank analysis having DMCs out of specification, the reviewer must give special consideration to the validity of associated sample data. The basic concern is whether the blank problems represent an isolated problem with the blank alone, or whether there is a fundamental problem with the analytical process. For example, if one or more samples in the batch show acceptable DMC recoveries, the reviewer may choose to consider the blank problem to be an isolated occurrence. However, even if this judgment allows some use of the affected data, note analytical problems for Contract Laboratory COR action.
- If more than three DMCs are outside of the recovery limits for Low/Medium volatiles analysis and the sample was not reanalyzed, note under Contract Problems/Non-Compliance.

**Table. Deuterated Monitoring Compound (DMC) Recovery Actions for Low/Medium Volatiles Analyses – Summary**

Criteria	Action	
	Detect Associated Compounds	Non-detected Associated Compounds
$\%R < 10\%$	J-	R
$10\% \leq \%R < \text{Lower Acceptance Limit}$	J-	UJ
$\text{Lower Acceptance Limit} \leq \%R \leq \text{Upper Acceptance Limit}$	No qualification	No qualification
$\%R > \text{Upper Acceptance Limit}$	J+	No qualification

DATA REVIEW WORKSHEETS

TABLE. VOLATILE DEUTERATED MONITORING COMPOUNDS (DMCs) AND THE ASSOCIATED TARGET COMPOUNDS

<b>Vinyl chloride-d<sub>3</sub> (DMC-1)</b>	<b>Chloroethane-d<sub>3</sub> (DMC-2)</b>	<b>1,1-Dichloroethene-d<sub>2</sub> (DMC-3)</b>
Vinyl chloride	Dichlorodifluoromethane Chloromethane Bromomethane Chloroethane Carbon disulfide	trans-1,2-Dichloroethene cis-1,2-Dichloroethene 1,1-Dichloroethene
<b>2-Butanone-d<sub>6</sub> (DMC-4)</b>	<b>Chloroform-d (DMC-5)</b>	<b>1,2-Dichloroethane-d<sub>2</sub> (DMC-6)</b>
Acetone 2-Butanone	1,1-Dichloroethane Bromochloromethane Chloroform Dibromochloromethane Bromoform	Trichlorofluoromethane 1,1,2-Trichloro-1,2,2-trifluoroethane Methyl acetate Methylene chloride Methyl-tert-butyl ether 1,1,1-Trichloroethane Carbon tetrachloride 1,2-Dibromoethane 1,2-Dichloroethane
<b>Benzene-d<sub>6</sub> (DMC-7)</b>	<b>1,2-Dichloropropane-d<sub>2</sub> (DMC-8)</b>	<b>Toluene-d<sub>8</sub> (DMC-9)</b>
Benzene	Cyclohexane Methylcyclohexane 1,2-Dichloropropane Bromodichloromethane	Trichloroethene Toluene Tetrachloroethene Ethylbenzene o-Xylene m,p-Xylene Styrene Isopropylbenzene
<b>trans-1,3-Dichloropropene-d<sub>1</sub> (DMC-10)</b>	<b>2-Hexanone-d<sub>8</sub> (DMC-11)</b>	<b>1,1,2,2-Tetrachloroethane-d<sub>2</sub> (DMC-12)</b>
cis-1,3-Dichloropropene trans-1,3-Dichloropropene 1,1,2-Trichloroethane	4-Methyl-2-pentanone 2-Hexanone	1,1,2,2-Tetrachloroethane 1,2-Dibromo-3-chloropropane
<b>1,2-Dichlorobenzene-d<sub>4</sub> (DMC-13)</b>		
Chlorobenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene 1,2-Dichlorobenzene 1,2,4-Trichlorobenzene 1,2,3-Trichlorobenzene		

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met \_\_\_\_\_  
 and/or see below \_\_\_\_\_

### MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples. If any % R in the MS or MSD falls outside the designated range, the reviewer should determine if there are matrix effects, i.e. LCS data are within the QC limits but MS/MSD data are outside QC limit.

**NOTES:** Data for MS and MSDs will not be present unless requested by the Region. Notify the Contract Laboratory COR if a field or trip blank was used for the MS and MSD.

For a Matrix Spike that does not meet criteria, apply the action to only the field sample used to prepare the Matrix Spike sample. If it is clearly stated in the data validation materials that the samples were taken through incremental sampling or some other method guaranteeing the homogeneity of the sample group, then the entire sample group may be qualified.

#### 1. MS/MSD Recoveries and Precision Criteria

The laboratory should use one MS and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If target analytes are not expected, MS/MSD should be analyzed.

List the %Rs, RPD of the compounds which do not meet the criteria.

Sample ID: FA41854-2MS/-2MSD

Matrix/Level: Groundwater

The QC reported here applies to the following samples:

Method: **SW846 8260C**

**FA41854-1, FA41854-2, FA41854-3**

Compound	FA41854-2 ug/l	Q	Spike ug/l	MS ug/	MS %	Spike ug/l	MSD ug/l	MSD %	RPD	Limits Rec/RPD
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**Note:** MS/MSD % recoveries and RPD within laboratory control limits.

Note:

- \* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- \* If QC limits are not available, use limits of 70 – 130 %.

## DATA REVIEW WORKSHEETS

### Actions:

1. No qualification of the data is necessary on MS and MSD data alone. However, using professional judgment, the validator may use the MS and MSD results in conjunction with other QC criteria and determine the need for some qualification of the data.

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

MS/MSD criteria apply only to the unspiked sample, its dilutions, and the associated MS/MSD samples:

If the % R for the affected compounds were < LL (or 70 %), qualify positive results (J) and nondetects (UJ).

If the % R for the affected compounds were > UL (or 130 %), only qualify positive results (J).

If 25 % or more of all MS/MSD %R were < LL (or 70 %) or if two or more MS/MSD %Rs were < 10%, qualify all positive results (J) and reject nondetects (R).

A separate worksheet should be used for each MS/MSD pair.

## DATA REVIEW WORKSHEETS

All criteria were met X  
 Criteria were not met  
 and/or see below \_\_\_\_\_

### LABORATORY CONTROL SAMPLE (LCS) ANALYSIS

This data is generated to determine accuracy of the analytical method for various matrices.

#### 1. LCS Recoveries Criteria

Where LCS spiked with the same analyte at the same concentrations as the MS/MSD? **Yes** or **No**. If no make note in data review memo.

List the %R of compounds which do not meet the criteria

LCS ID	COMPOUND	% R	QC LIMIT
Recoveries (blank spike) within laboratory control limits. _____			
_____			
_____			
_____			
_____			

#### Note:

- \* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- \* If QC limits are not available, use limits of 70 – 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

All analytes in the associated sample results are qualified for the following criteria.

If 25 % of the LCS recoveries were < LL (or 70 %), qualify all positive results (j) and reject nondetects (R).

If two or more LCS were below 10 %, qualify all positive results as (J) and reject nondetects (R).

#### 2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix? **Yes** or **No**.

If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected.

## DATA REVIEW WORKSHEETS

All criteria were met   N/A    
Criteria were not met  
and/or see below                     

### IX. FIELD/LABORATORY DUPLICATE PRECISION

Sample IDs:                      -                     

Matrix:                      -                     

Field/laboratory duplicate samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

The project QAPP should be reviewed for project-specific information.

**NOTE:** In the absence of QAPP guidance for validating data from field duplicates, the following action will be taken.

Identify which samples within the data package are field duplicates. Estimate the relative percent difference (RPD) between the values for each compound. Use professional judgment to note large RPDs (> 50%) in the narrative.

COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION
No field/laboratory duplicate analyzed with this data package. MS/MSD % recovery RPD used to assess precision. RPD within required criteria, $\leq 50\%$ for target analytes detected at concentration > 5x the SQL.					

#### Actions:

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria. For organics, only the sample and duplicate will be qualified.

If an RPD cannot be calculated because one or both of the sample results is not detected, the following actions are suggested based on professional judgment:

If one sample result is not detected and the other is greater than 5x the SQL qualify (J/UJ).

If one sample value is not detected and the other is greater than 5x the SQL and the SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is less than 5x, use professional judgment to determine if qualification is appropriate.

If both sample and duplicate results are not detected, no action is needed.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met  
 and/or see below           

### X. INTERNAL STANDARD PERFORMANCE

The assessment of the internal standard (IS) parameter is used to assist the data reviewer in determining the condition of the analytical instrumentation.

DATE	SAMPLE ID	IS OUT	IS AREA	ACCEPTABLE RANGE	ACTION
Internal standard area within laboratory control limits.					

#### Action:

1. If an internal standard area count for a sample or blank is greater than 200.0% of the area for the associated standard (opening CCV or mid-point standard from initial calibration) (see Table below):
  - a. Qualify detects for compounds quantitated using that internal standard as estimated low (J-).
  - b. Do not qualify non-detected associated compounds.
2. If an internal standard area count for a sample or blank is less than 20.0% of the area for the associated standard (opening CCV or mid-point standard from initial calibration):
  - a. Qualify detects for compounds quantitated using that internal standard as estimated high (J+).
  - b. Qualify non-detected associated compounds as unusable (R).
3. If an internal standard area count for a sample or blank is greater than or equal to 20.0%, and less than or equal to 200% of the area for the associated standard opening CCV or mid-point standard from initial calibration, no qualification of the data is necessary.
4. If an internal standard RT varies by more than 30.0 seconds: Examine the chromatographic profile for that sample to determine if any false positives or negatives exist. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for that sample fraction. Detects should not need to be qualified as unusable (R) if the mass spectral criteria are met.
5. If an internal standard RT varies by less than or equal to 30.0 seconds, no qualification of the data is necessary.

**Note:** Inform the Contract Laboratory Program Project Officer (CLP PO) if the internal standard performance criteria are grossly exceeded. Note in the Data Review Narrative potential effects on the data resulting from unacceptable internal standard performance.



## DATA REVIEW WORKSHEETS

6. If required internal standard compounds are not added to a sample or blank, qualify detects and non-detects as unusable (R).
7. If the required internal standard compound is not analyzed at the specified concentration in a sample or blank, use professional judgment to qualify detects and non-detects.

**Table. Internal Standard Actions for Low/Medium Volatiles Analyses - Summary**

Criteria	Action	
	Detected Associated Compounds*	Non-detected Associated Compounds*
Area counts > 200% of 12-hour standard (opening CCV or mid-point standard from initial calibration)	J-	No qualification
Area counts < 20% of 12-hour standard (opening CCV or mid-point standard from initial calibration)	J+	R
Area counts ≥ 50% but ≤ 200% of 12-hour standard (opening CCV or mid-point standard from initial calibration)	No qualification	
RT difference > 30.0 seconds between samples and 12-hour standard (opening CCV or mid-point standard from initial calibration)	R **	R
RT difference ≤ 30.0 seconds between samples and 12-hour standard (opening CCV or mid-point standard from initial calibration)	No qualification	

\* For volatile compounds associated to each internal standard, see TABLE - VOLATILE TARGET ANALYTES, DEUTERATED MONITORING COMPOUNDS WITH ASSOCIATED INTERNAL STANDARDS FOR QUANTITATION in SOM02.2, Exhibit D, available at: <http://www.epa.gov/superfund/programs/clp/download/som/som22d.pdf>

\*\* Detects should not need to be qualified as unusable (R) if the mass spectral criteria are met.

## DATA REVIEW WORKSHEETS

All criteria were met X  
 Criteria were not met  
 and/or see below \_\_\_\_\_

### TARGET COMPOUND IDENTIFICATION

#### Criteria:

Is the Relative Retention Times (RRTs) of reported compounds within  $\pm 0.06$  RRT units of the standard RRT [opening Continuing Calibration Verification (CCV) or mid-point standard from the initial calibration]. Yes? or No?

List compounds not meeting the criteria described above:

Sample ID	Compounds	Actions
=====	=====	=====
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

Mass spectra of the sample compound and a current laboratory-generated standard [i.e., the mass spectrum from the associated calibration standard (opening CCV or mid-point standard from initial calibration)] must match according to the following criteria:

- All ions present in the standard mass spectrum at a relative intensity greater than 10% must be present in the sample spectrum.
- The relative intensities of these ions must agree within  $\pm 20\%$  between the standard and sample spectra (e.g., for an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30-70%).
- Ions present at greater than 10% in the sample mass spectrum, but not present in the standard spectrum, must be evaluated by a reviewer experienced in mass spectral interpretation.

List compounds not meeting the criteria described above:

Sample ID	Compounds	Actions
=====	=====	=====
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

## DATA REVIEW WORKSHEETS

### Action:

1. The application of qualitative criteria for GC/MS analysis of target compounds requires professional judgment. It is up to the reviewer's discretion to obtain additional information from the laboratory. If it is determined that incorrect identifications were made, qualify all such data as unusable (R).
2. Use professional judgment to qualify the data if it is determined that cross-contamination has occurred.
3. Note in the Data Review Narrative any changes made to the reported compounds or concerns regarding target compound identifications. Note, for Contract Laboratory COR action, the necessity for numerous or significant changes.

### TENTATIVELY IDENTIFIED COMPOUNDS (TICS)

NOTE: Tentatively identified compounds should only be evaluated when requested by a party from outside of the Hazardous Waste Support Section (HWSS).

#### List TICs

Sample ID	Compound	Sample ID	Compound
=====			

### Action:

1. Qualify all TIC results for which there is presumptive evidence of a match (e.g. greater than or equal to 85% match) as tentatively identified (NJ), with approximated concentrations. TICs labeled "unknown" are qualified as estimated (J).
2. General actions related to the review of TIC results are as follows:
  - a. If it is determined that a tentative identification of a non-target compound is unacceptable, change the tentative identification to "unknown" or another appropriate identification, and qualify the result as estimated (J).
  - b. If all contractually-required peaks were not library searched and quantitated, the Region's designated representative may request these data from the laboratory.
3. In deciding whether a library search result for a TIC represents a reasonable identification, use professional judgment. If there is more than one possible match, report the result as "either compound X or compound Y". If there is a lack of isomer specificity, change the TIC result to a nonspecific isomer result (e.g., 1,3,5-trimethyl benzene to trimethyl benzene).

## DATA REVIEW WORKSHEETS

- isomer) or to a compound class (e.g., 2-methyl, 3-ethyl benzene to a substituted aromatic compound).
4. The reviewer may elect to report all similar compounds as a total (e.g., all alkanes may be summarized and reported as total hydrocarbons).
  5. Target compounds from other fractions and suspected laboratory contaminants should be marked as "non-reportable".
  6. Other Case factors may influence TIC judgments. If a sample TIC match is poor, but other samples have a TIC with a valid library match, similar RRT, and the same ions, infer identification information from the other sample TIC results.
  7. Note in the Data Review Narrative any changes made to the reported data or any concerns regarding TIC identifications.
  8. Note, for Contract Laboratory COR action, failure to properly evaluate and report TICs

## DATA REVIEW WORKSHEETS

All criteria were met X  
 Criteria were not met  
 and/or see below \_\_\_\_\_

### SAMPLE QUANTITATION AND REPORTED CONTRACT REQUIRED QUANTITATION LIMITS (CRQLS)

#### Action:

1. If any discrepancies are found, the Region's designated representative may contact the laboratory to obtain additional information that could resolve any differences. If a discrepancy remains unresolved, the reviewer must use professional judgment to decide which value is the most accurate. Under these circumstances, the reviewer may determine that qualification of data is warranted. Note in the Data Review Narrative a description of the reasons for data qualification and the qualification that is applied to the data.
2. For non-aqueous samples, if the percent moisture is less than 70.0%, no qualification of the data is necessary. If the percent moisture is greater than or equal to 70.0% and less than 90.0%, qualify detects as estimated (J) and non-detects as approximated (UJ). If the percent moisture is greater than or equal to 90.0%, qualify detects as estimated (J) and non-detects as unusable (R) (see Table below).
3. Note, for Contract Laboratory COR action, numerous or significant failures to accurately quantify the target compounds or to properly evaluate and adjust CRQLs.
4. Results between MDL and CRQL should be qualified as estimated "J".
5. Results < MDL should be reported at the CRQL and qualified "U". MDLs themselves are not reported.

Table. Percent Moisture Actions for Low/Medium Volatiles Analysis for Non-Aqueous Samples

Criteria	Action	
	Detected Associated Compounds	Non-detected Associated Compounds
% Moisture < 70.0	No qualification	
70.0 < % Moisture < 90.0	J	UJ
% Moisture > 90.0	J	R

The sample quantitation evaluation is to verify laboratory quantitation results. In the space below, please show a minimum of one sample calculation:

Sample ID

FA41854-2MS

Chloroform

RF = 0.484

[ ] = (556309)/(50)/(0.484)(2263090) = 25.39 ppb Ok

• • • • •

All criteria were met X  
Criteria were not met  
and/or see below \_\_\_\_\_

### B. Percent Solids

List samples which have  $\geq 70\%$  solids

*(The following section contains faint, illegible markings, possibly bleed-through from the reverse side of the page.)*

## QUANTITATION LIMITS

A. Dilution performed

[illegible]

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met  
 and/or see below       

### OTHER ISSUES

#### A. System Performance

List samples qualified based on the degradation of system performance during sample analysis:

Sample ID	Comments	Actions
=====		
<u>      </u>	<u>      </u>	<u>      </u>
<u>No_degradation_of_system_performance_observed.</u>	<u>      </u>	<u>      </u>
<u>      </u>	<u>      </u>	<u>      </u>
<u>      </u>	<u>      </u>	<u>      </u>

Action:

Use professional judgment to qualify the data if it is determined that system performance has degraded during sample analyses. Inform the Contract Laboratory Program COR any action as a result of degradation of system performance which significantly affected the data.

#### B. Overall Assessment of Data

List samples qualified based on other issues:

Sample ID	Comments	Actions
=====		
<u>      </u>	<u>      </u>	<u>      </u>
<u>No_additional_issues_observed_that_require_qualification_of_the_data._Results_are_valid_and</u>	<u>      </u>	<u>      </u>
<u>can_be_used_for_decision_purposes.</u>	<u>      </u>	<u>      </u>
<u>      </u>	<u>      </u>	<u>      </u>
<u>      </u>	<u>      </u>	<u>      </u>

Action:

1. Use professional judgment to determine if there is any need to qualify data which were not qualified based on the Quality Control (QC) criteria previously discussed.
2. Write a brief narrative to give the user an indication of the analytical limitations of the data. Inform the Contract Laboratory COR the action, any inconsistency of the data with the Sample Delivery Group (SDG) Narrative. If sufficient information on the intended use and required quality of the data is available, the reviewer should include their assessment of the usability of the data within the given context. This may be used as part of a formal Data Quality Assessment (DQA).

## EXECUTIVE NARRATIVE

SDG No: FA41854 Laboratory: Accutest, Orlando  
Analysis: SW846-8270D Number of Samples: 4  
Location: BMSMC, Humacao, PR

**SUMMARY:** Four (4) samples were analyzed for Benzaldehyde and bis(2-Ethylhexyl)phthalate following method SW846-8270D; Selected PAHs and 1,4-Dioxane were also analyzed by SW846-8270D using the selective ion monitoring (SIM) technique; samples were analyzed separately for each analyte group. The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: EPA Hazardous Waste Support Section, SOP HW-35A, July 2015 –Revision 0. Semivolatile Data Validation. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

Results are valid and can be used for decision making purposes.

**Critical issues:** None  
**Major:** None  
**Minor:** None

**Critical findings:** None  
**Major findings:** None

**Minor findings:** 1. No closing calibration verification included in data package for instruments GCMSV and GCMSX. No action taken, professional judgment.

2. MS/MSD % recoveries and RPD within laboratory control limits except for the cases described in the Data Review Worksheet. MS/MSD % recovery for bis(2-Ethylhexyl)phthalate outside laboratory control limits. No action taken, professional judgment. Analyte recovered high and not detected in sample batch.

3. bis(2-Ethylhexyl)phthalate recover high in Blank Spike. No action taken, professional judgment; bis(2-Ethylhexyl)phthalate not detected in sample batch.

**COMMENTS:** Results are valid and can be used for decision making purposes.

**Reviewers Name:** Rafael Infante  
Chemist License 1888

**Signature:** \_\_\_\_\_  
**Date:** April 14 2017



# ORGANIC DATA SAMPLE SUMMARY

Sample ID: FA41854-1

Sample location: BMSMC, Humacao, PR

Sampling date: 3/6/2017

Matrix: Groundwater

## METHOD: 8270D

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Benzaldehyde	24	ug/L	1.0	-	U	Yes
bis(2-Ethylhexyl)phthalate	4.8	ug/L	1.0	-	U	Yes

## METHOD: 8270D SIM

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Benzo(a)anthracene	0.19	ug/L	1.0	-	U	Yes
1,4-Dioxane	0.72	ug/L	1.0	-	-	Yes
Naphthalene	0.95	ug/L	1.0	-	U	Yes

Sample ID: FA41854-2

Sample location: BMSMC, Humacao, PR

Sampling date: 3/6/2017

Matrix: Groundwater

## METHOD: 8270D

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Benzaldehyde	24	ug/L	1.0	-	U	Yes
bis(2-Ethylhexyl)phthalate	4.8	ug/L	1.0	-	U	Yes

## METHOD: 8270D SIM

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Benzo(a)anthracene	0.19	ug/L	1.0	-	U	Yes
1,4-Dioxane	1.8	ug/L	1.0	-	-	Yes
Naphthalene	0.95	ug/L	1.0	-	U	Yes

Sample ID: FA41854-2MS

Sample location: BMSMC, Humacao, PR

Sampling date: 3/6/2017

Matrix: Groundwater

METHOD: 8270D

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Benzaldehyde	91.1	ug/L	1.0	-	-	Yes
bis(2-Ethylhexyl)phthalate	142	ug/L	1.0	-	-	Yes

METHOD: 8270D SIM

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
1,4-Dioxane	5.0	ug/L	1.0	-	-	Yes

METHOD: 8270D SIM

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Benzo(a)anthracene	9.3	ug/L	1.0	-	-	Yes
Naphthalene	16.4	ug/L	1.0	-	-	Yes

Sample ID: FA41854-2MSD

Sample location: BMSMC, Humacao, PR

Sampling date: 3/6/2017

Matrix: Groundwater

METHOD: 8270D

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Benzaldehyde	91.2	ug/L	1.0	-	-	Yes
bis(2-Ethylhexyl)phthalate	133	ug/L	1.0	-	-	Yes

METHOD: 8270D SIM

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
1,4-Dioxane	5.1	ug/L	1.0	-	-	Yes

METHOD: 8270D SIM

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Benzo(a)anthracene	9.2	ug/L	1.0	-	-	Yes
Naphthalene	16.1	ug/L	1.0	-	-	Yes

# DATA REVIEW WORKSHEETS

Project Number: FA41854  
 Date: March 6, 2017  
 Shipping Date: March 7, 2017  
 EPA Region: 2

## REVIEW OF SEMIVOLATILE ORGANIC PACKAGE

The following guidelines for evaluating volatile organics were created to delineate required validation actions. This document will assist the reviewer in using professional judgment to make more informed decision and in better serving the needs of the data users. The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: EPA Hazardous Waste Support Section, SOP HW-35A, July 2015 –Revision 0. *Semivolatile Data Validation*. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

The hardcopied (laboratory name) Accutest data package received has been reviewed and the quality control and performance data summarized. The data review for SVOCs included:

Lab. Project/SDG No.: FA41854 Sample matrix: Groundwater  
 No. of Samples: 8 SIM/4 SCAN  
 Trip blank No.: -  
 Field blank No.: -  
 Equipment blank No.: -  
 Field duplicate No.: -

<input checked="" type="checkbox"/> Data Completeness	<input checked="" type="checkbox"/> Laboratory Control Spikes
<input checked="" type="checkbox"/> Holding Times	<input checked="" type="checkbox"/> Field Duplicates
<input checked="" type="checkbox"/> GC/MS Tuning	<input checked="" type="checkbox"/> Calibrations
<input checked="" type="checkbox"/> Internal Standard Performance	<input checked="" type="checkbox"/> Compound Identifications
<input checked="" type="checkbox"/> Blanks	<input checked="" type="checkbox"/> Compound Quantitation
<input checked="" type="checkbox"/> Surrogate Recoveries	<input checked="" type="checkbox"/> Quantitation Limits
<input checked="" type="checkbox"/> Matrix Spike/Matrix Spike Duplicate	

Overall Comments: Selected SVOCs from the TCL special list analyzed by method SW846-8270D; Selected PAHs and 1,4-Dioxane analyzed by method SW846-8270D (SIM); 1,4-dioxane and PAH's analyzed separately.

### Definition of Qualifiers:

J- Estimated results  
 U- Compound not detected  
 R- Rejected data  
 UJ- Estimated nondetect

Reviewer: Rafael Infante  
 Date: April 14, 2017



## DATA REVIEW WORKSHEETS

All criteria were met X  
 Criteria were not met  
 and/or see below \_\_\_\_\_

### HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE EXTRACTED/ANALYZED	pH	ACTION
All samples extracted and analyzed within method recommended holding time. Sample preservation appropriate.				

Cooler temperature (Criteria:  $4 \pm 2$  °C): 3.0/3.2 °C \_\_\_\_\_

#### Actions

Results will be qualified based on the criteria of the following Table:

**Table 1. Holding Time Actions for Semivolatile Analyses**

Matrix	Preserved	Criteria	Action	
			Detected Associated Compounds	Non-Detected Associated Compounds
Aqueous	No	$\leq 7$ days (for extraction) $\leq 40$ days (for analysis)	Use professional judgment	
	No	$> 7$ days (for extraction) $> 40$ days (for analysis)	J	Use professional judgment
	Yes	$\leq 7$ days (for extraction) $\leq 40$ days (for analysis)	No qualification	
	Yes	$> 7$ days (for extraction) $> 40$ days (for analysis)	J	UJ
	Yes/No	Grossly Exceeded	J	UJ or R
Non-Aqueous	No	$\leq 14$ days (for extraction) $\leq 40$ days (for analysis)	Use professional judgment	
	No	$> 14$ days (for extraction) $> 40$ days (for analysis)	J	Use professional judgment
	Yes	$\leq 14$ days (for extraction) $\leq 40$ days (for analysis)	No qualification	
	Yes	$> 14$ days (for extraction) $> 40$ days (for analysis)	J	UJ
	Yes/No	Grossly Exceeded	J	UJ or R

All criteria were met X  
 Criteria were not met see below \_\_\_\_\_

## DATA REVIEW WORKSHEETS

### GC/MS TUNING

The assessment of the tuning results is to determine if the sample instrumentation is within the standard tuning QC limits

☒ The DFTPP performance results were reviewed and found to be within the specified criteria.

☒ DFTPP tuning was performed for every 12 hours of sample analysis.

If no, use professional judgment to determine whether the associated data should be accepted, qualified or rejected.

Notes: These requirements do not apply when samples are analyzed by the Selected Ion Monitoring (SIM) technique.

All mass spectrometer conditions must be identical to those used during the sample analysis. Background subtraction actions resulting in spectral distortion are unacceptable

Notes: No data should be qualified based of DFTPP failure.

The requirement to analyze the instrument performance check solution is optional when analysis of PAHs/pentachlorophenol is to be performed by the SIM technique.

List	the	samples	affected:
<hr/>			
<hr/>			
<hr/>			
<hr/>			

#### Actions:

1. If sample are analyzed without a preceding valid instrument performance check or are analyzed 12 hours after the Instrument Performance Check, qualify all data in those samples as unusable (R).
2. If ion abundance criteria are not met, use professional judgment to determine to what extent the data may be utilized.
3. State in the Data Review Narrative, decisions to use analytical data associated with DFTPP instrument performance checks not meeting the contract requirements.
4. Use professional judgment to determine if associated data should be qualified based on the spectrum of the mass calibration compounds.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met  
 and/or see below           

### INITIAL CALIBRATION VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration:   02/13/17   (SIM)                      03/13/17   (SCAN)                   

Instrument ID numbers:   GCMSW                        GCMSX                     

Matrix/Level:   Aqueous/low                        Aqueous/low                     

Date of initial calibration:   02/23/17   (SIM)                   

Instrument ID numbers:   GCMSV                     

Matrix/Level:   Aqueous/low                     

DATE	LAB ID#	FILE	CRITERIA OUT RFs, %RSD, %D, r	COMPOUND	SAMPLES AFFECTED
Initial and initial calibration verification meets the method and guidance validation document performance criteria.					

**Note:**

**Actions:**

Qualify the initial calibration analytes listed in Table 2 using the following criteria:

**Table 3. Initial Calibration Actions for Semivolatile Analysis**

Criteria	Action	
	Detect	Non-detect
Initial Calibration not performed at specified frequency and sequence	Use professional judgment R	Use professional judgment R
Initial Calibration not performed at the specified concentrations	J	UJ
RRF < Minimum RRF in Table 2 for target analyte	Use professional judgment J+ or R	R
RRF ≥ Minimum RRF in Table 2 for target analyte	No qualification	No qualification
%RSD > Maximum %RSD in Table 2 for target analyte	J	Use professional judgment
%RSD ≤ Maximum %RSD in Table 2 for target analyte	No qualification	No qualification



## DATA REVIEW WORKSHEETS

### Initial Calibration

**Table 2. RRF, %RSD, and %D Acceptance Criteria in Initial Calibration and CCV for Semivolatil Analysis**

Analyte	Minimum RRF	Maximum %RSD	Opening Maximum %D <sup>1</sup>	Opening Maximum %D <sup>1</sup>
1,4-Dioxane	0.010	40.0	± 40.0	± 50.0
Benzaldehyde	0.100	40.0	± 40.0	± 50.0
Phenol	0.080	20.0	± 20.0	± 25.0
Bis(2-chloroethyl)ether	0.100	20.0	± 20.0	± 25.0
2-Chlorophenol	0.200	20.0	± 20.0	± 25.0
2-Methylphenol	0.010	20.0	± 20.0	± 25.0
3-Methylphenol	0.010	20.0	± 20.0	± 25.0
2,2'-(Oxybis-(1-chloropropane))	0.010	20.0	± 25.0	± 50.0
Acetophenone	0.060	20.0	± 20.0	± 25.0
4-Methylphenol	0.010	20.0	± 20.0	± 25.0
N-Nitroso-di-n-propylamine	0.080	20.0	± 25.0	± 25.0
Hexachloroethane	0.100	20.0	± 20.0	± 25.0
Nitrobenzene	0.090	20.0	± 20.0	± 25.0
Isophorone	0.100	20.0	± 20.0	± 25.0
2-Nitrophenol	0.060	20.0	± 20.0	± 25.0
2,4-Dimethylphenol	0.050	20.0	± 25.0	± 50.0
Bis(2-chloroethoxy)methane	0.080	20.0	± 20.0	± 25.0
2,4-Dichlorophenol	0.060	20.0	± 20.0	± 25.0
Naphthalene	0.200	20.0	± 20.0	± 25.0
4-Chloroaniline	0.010	40.0	± 40.0	± 50.0
Hexachlorobutadiene	0.040	20.0	± 20.0	± 25.0
Caprolactam	0.010	40.0	± 30.0	± 50.0
4-Chloro-3-methylphenol	0.040	20.0	± 20.0	± 25.0
2-Methylnaphthalene	0.100	20.0	± 20.0	± 25.0
Hexachlorocyclopentadiene	0.010	40.0	± 40.0	± 50.0
2,4,6-Trichlorophenol	0.090	20.0	± 20.0	± 25.0
2,4,5-Trichlorophenol	0.100	20.0	± 20.0	± 25.0
1,1'-Biphenyl	0.200	20.0	± 20.0	± 25.0

DATA REVIEW WORKSHEETS

Analyte	Minimum RRF	Maximum %RSD	Opening Maximum %D <sup>1</sup>	Opening Maximum %D <sup>1</sup>
2-Chloronaphthalene	0.300	20.0	± 20.0	± 25.0
2-Nitroaniline	0.060	20.0	± 25.0	± 25.0
Dimethylphthalate	0.300	20.0	± 25.0	± 25.0
2,6-Dinitrotoluene	0.080	20.0	± 20.0	± 25.0
Acenaphthylene	0.400	20.0	± 20.0	± 25.0
3-Nitroaniline	0.010	20.0	± 25.0	± 50.0
Acenaphthene	0.200	20.0	± 20.0	± 25.0
2,4-Dinitrophenol	0.010	40.0	± 50.0	± 50.0
4-Nitrophenol	0.010	40.0	± 40.0	± 50.0
Dibenzofuran	0.300	20.0	± 20.0	± 25.0
2,4-Dinitrotoluene	0.070	20.0	± 20.0	± 25.0
Diethylphthalate	0.300	20.0	± 20.0	± 25.0
1,2,4,5-Tetrachlorobenzene	0.100	20.0	± 20.0	± 25.0
4-Chlorophenyl-phenylether	0.100	20.0	± 20.0	± 25.0
Fluorene	0.200	20.0	± 20.0	± 25.0
4-Nitroaniline	0.010	40.0	± 40.0	± 50.0
4,6-Dinitro-2-methylphenol	0.010	40.0	± 30.0	± 50.0
4-Bromophenyl-phenyl ether	0.070	20.0	± 20.0	± 25.0
N-Nitrosodiphenylamine	0.100	20.0	± 20.0	± 25.0
Hexachlorobenzene	0.050	20.0	± 20.0	± 25.0
Atrazine	0.010	40.0	± 25.0	± 50.0
Pentachlorophenol	0.010	40.0	± 40.0	± 50.0
Phenanthrene	0.200	20.0	± 20.0	± 25.0
Anthracene	0.200	20.0	± 20.0	± 25.0
Carbazole	0.050	20.0	± 20.0	± 25.0
Di-n-butylphthalate	0.500	20.0	± 20.0	± 25.0
Fluoranthene	0.100	20.0	± 20.0	± 25.0
Pyrene	0.400	20.0	± 25.0	± 50.0
Butylbenzylphthalate	0.100	20.0	± 25.0	± 50.0

DATA REVIEW WORKSHEETS

Analyte	Minimum RRF	Maximum %RSD	Opening Maximum %D <sup>1</sup>	Opening Maximum %D <sup>1</sup>
3,3'-Dichlorobenzidine	0.010	40.0	± 40.0	± 50.0
Benzo(a)anthracene	0.300	20.0	± 20.0	± 25.0
Chrysene	0.200	20.0	± 20.0	± 50.0
Bis(2-ethylhexyl) phthalate	0.200	20.0	± 25.0	± 50.0
Di-n-octylphthalate	0.010	40.0	± 40.0	± 50.0
Benzo(b)fluoranthene	0.010	20.0	± 25.0	± 50.0
Benzo(k)fluoranthene	0.010	20.0	± 25.0	± 50.0
Benzo(a)pyrene	0.010	20.0	± 20.0	± 50.0
Indeno(1,2,3-cd)pyrene	0.010	20.0	± 25.0	± 50.0
Dibenzo(a,h)anthracene	0.010	20.0	± 25.0	± 50.0
Benzo(g,h,i)perylene	0.010	20.0	± 30.0	± 50.0
2,3,4,6-Tetrachlorophenol	0.040	20.0	± 20.0	± 50.0
Naphthalene	0.600	20.0	± 25.0	± 25.0
2-Methylnaphthalene	0.300	20.0	± 20.0	± 25.0
Acenaphthylene	0.900	20.0	± 20.0	± 25.0
Acenaphthene	0.500	20.0	± 20.0	± 25.0
Fluorene	0.700	20.0	± 25.0	± 50.0
Phenanthrene	0.300	20.0	± 25.0	± 50.0
Anthracene	0.400	20.0	± 25.0	± 50.0
Fluoranthene	0.400	20.0	± 25.0	± 50.0
Pyrene	0.500	20.0	± 30.0	± 50.0
Benzo(a)anthracene	0.400	20.0	± 25.0	± 50.0
Chrysene	0.400	20.0	± 25.0	± 50.0
Benzo(b)fluoranthene	0.100	20.0	± 30.0	± 50.0
Benzo(k)fluoranthene	0.100	20.0	± 30.0	± 50.0
Benzo(a)pyrene	0.100	20.0	± 25.0	± 50.0
Indeno(1,2,3-cd)pyrene	0.100	20.0	± 40.0	± 50.0
Dibenzo(a,h)anthracene	0.010	25.0	± 40.0	± 50.0
Benzo(g,h,i)perylene	0.020	25.0	± 40.0	± 50.0

# DATA REVIEW WORKSHEETS

Pentachlorophenol	0.010	40.0	± 50.0	± 50.0
<b>Deuterated Monitoring Compounds</b>				
Analyte	Minimum RRF	Maximum %RSD	Opening Maximum %D <sup>1</sup>	Closing Maximum %D
1,4-Dioxane-d <sub>8</sub>	0.010	20.0	± 25.0	± 50.0
Phenol-d <sub>5</sub>	0.010	20.0	± 25.0	± 25.0
Bis-(2-chloroethyl)ether-d <sub>8</sub>	0.100	20.0	± 20.0	± 25.0
2-Chlorophenol-d <sub>4</sub>	0.200	20.0	± 20.0	± 25.0
4-Methylphenol-d <sub>8</sub>	0.010	20.0	± 20.0	± 25.0
4-Chloroaniline-d <sub>4</sub>	0.010	40.0	± 40.0	± 50.0
Nitrobenzene-d <sub>5</sub>	0.050	20.0	± 20.0	± 25.0
2-Nitrophenol-d <sub>4</sub>	0.050	20.0	± 20.0	± 25.0
2,4-Dichlorophenol-d <sub>3</sub>	0.060	20.0	± 20.0	± 25.0
Dimethylphthalate-d <sub>6</sub>	0.300	20.0	± 20.0	± 25.0
Acenaphthylene-d <sub>8</sub>	0.400	20.0	± 20.0	± 25.0
4-Nitrophenol-d <sub>4</sub>	0.010	40.0	± 40.0	± 50.0
Fluorene-d <sub>10</sub>	0.100	20.0	± 20.0	± 25.0
4,6-Dinitro-2-methylphenol-d <sub>2</sub>	0.010	40.0	± 30.0	± 50.0
Anthracene-d <sub>10</sub>	0.300	20.0	± 20.0	± 25.0
Pyrene-d <sub>10</sub>	0.300	20.0	± 25.0	± 50.0
Benzo(a)pyrene-d <sub>12</sub>	0.010	20.0	± 20.0	± 50.0
Fluoranthene-d <sub>10</sub> (SIM)	0.400	20.0	± 25.0	± 50.0
2-Methylnaphthalene-d <sub>10</sub> (SIM)	0.300	20.0	± 20.0	± 25.0

<sup>1</sup> If a closing CCV is acting as an opening CCV, all target analytes must meet the requirements for an opening CCV.

**Note:** If analysis by SIM technique is requested for PAH/pentachlorophenols, calibration standards analyzed at 0.10, 0.20, 0.40, 0.80, and 1.0 ng/uL for each target compound of interest and the associated DMCs. Pentachlorophenol will require only a four point initial calibration at 0.20, 0.40, 0.80, and 1.0 ng/uL.

## DATA REVIEW WORKSHEETS

All criteria were met X  
 Criteria were not met  
 and/or see below \_\_\_\_\_

### CONTINUING CALIBRATION VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration: 02/13/17 (SIM) 02/23/17 (SIM)  
 Date of initial calibration verification (ICV): 02/13/17 02/23/17  
 Date of continuing calibration verification (CCV): 03/17/17 03/14/17  
 Date of closing CCV: 02/14/17; 03/18/17 -  
 Instrument ID numbers: GCMSW GCMSV  
 Matrix/Level: Aqueous/low Aqueous/low

Date of initial calibration: 03/13/17 (SCAN)  
 Date of initial calibration verification (ICV): 03/13/17  
 Date of continuing calibration verification (CCV): 03/14/17  
 Date of closing CCV: -  
 Instrument ID numbers: GCMSX  
 Matrix/Level: Aqueous/low

DATE	LAB ID#	FILE	CRITERIA OUT RFs, %RSD, %D, r	COMPOUND	SAMPLES AFFECTED

**Note:** Initial and continuing calibration verifications meet the method and guidance document required performance criteria.

No closing calibration verification included in data package for instruments GCMSV (SIM) and GCMSX (SCAN). No action taken, professional judgment.

#### Actions:

**Notes:** Verify that the CCV is run at the required frequency (an opening and closing CCV must be run within 12-hour period).

All DMCs must meet the RRF values given in Table 2. No qualification of the data is necessary on DMCs RRF and %RSD/%D alone. Use professional judgment to evaluate DMCs and %RSD/%D data in conjunction with DMCs recoveries to determine the need for qualification of the data.

Qualify the initial calibration analytes listed in Table 2 using the following criteria in the CCVs:

## DATA REVIEW WORKSHEETS

**Table 4. CCV Actions for Semivolatile Analysis**

Criteria for Opening CCV	Criteria for Closing CCV	Action	
		Detect	Non-detect
CCV not performed at required frequency and sequence	CCV not performed at required frequency	Use professional judgment R	Use professional judgment R
CCV not performed at specified concentration	CCV not performed at specified concentration	Use professional judgment	Use professional judgment
$RRF \leq$ Minimum RRF in Table 2 for target analyte	$RRF \leq$ Minimum RRF in Table 2 for target analyte	Use professional judgment J or R	R
$RRF \geq$ Minimum RRF in Table 2 for target analyte	$RRF \geq$ Minimum RRF in Table 2 for target analyte	No qualification	No qualification
%D outside the Opening Maximum %D limits in Table 2 for target analyte	%D outside the Closing Maximum %D limits in Table 2 for target analyte	J	UJ
%D within the inclusive Opening Maximum %D limits in Table 2 for target analyte	%D within the inclusive Closing Maximum %D limits in Table 2 for target analyte	No qualification	No qualification

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met  
 and/or see below           

### BLANK ANALYSIS RESULTS (Sections 1 & 2)

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

List the contamination in the blanks below. High and low levels blanks must be treated separately.

Notes: The concentration of non-target compounds in all blanks must be less than or equal to 10 ug/L.

The concentration of target compounds in all blanks must be less than its CRQL listed in the method.

Samples taken from a drinking water tap do not have and associated field blank.

#### Laboratory blanks

DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
_No_target_analytes_detected_in_method_blanks_				

#### Field/Equipment/Trip blank

DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
_No_field/equipment_blanks_analyzed_with_this_data_package_				

**Note:**

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met  
 and/or see below           

### BLANK ANALYSIS RESULTS (Section 3)

#### Blank Actions

Qualify samples based on the criteria summarized in Table 5:

**Table 5. Blank and TCLP/SPLP LEB Actions for Semivolatile Analysis**

Blank Type	Blank Result	Sample Result	Action
Method, TCLP/SPLP LEB, Field	Detect	Non-detect	No qualification
	< CRQL	< CRQL	Report at CRQL and qualify as non-detect (U)
		≥ CRQL	Use professional judgment
	≥ CRQL	< CRQL	Report at CRQL and qualify as non-detect (U)
		≥ CRQL but < Blank Result	Report at sample results and qualify as non-detect (U) or as unusable (R)
		≥ CRQL and ≥ Blank Result	Use professional judgment
	Grossly high	Detect	Report at sample results and qualify as unusable (R)
	TIC > 5.0 ug/L (water) or 0.0050 mg/L (TCLP leachate) or TIC > 170 ug/Kg (soil)	Detect	Use professional judgment

List samples qualified

CONTAMINATION SOURCE/LEVEL	COMPOUND	CONC/UNITS	AL/UNITS	SQL	AFFECTED SAMPLES



## DATA REVIEW WORKSHEETS

All criteria were met \_\_\_\_\_  
 Criteria were not met \_\_\_\_\_  
 and/or see below   X  

### SURROGATE SPIKE RECOVERIES – DEUTERATED MONITORING COMPOUNDS (DMCs)

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries – deuterated monitoring compounds. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

Notes: Recoveries for DMCs in samples and blanks must be within the limits specified in Table 6.

The recovery limits for any of the compounds listed in Table 6 may be expanded at any time during the period of performance if USEPA determines that the limits are too restrictive.

If a DMC is not added in the samples and blanks or the concentrations of DMCs in the samples and blank not the specified, use professional judgment in qualifying the data.

**Table 7. DMC Actions for Semivolatile Analysis**

Criteria	Action	
	Detect	Non-detect
%R < 10% (excluding DMCs with 10% as a lower acceptance limit)	J-	R
10% ≤ %R (excluding DMCs with 10% as a lower acceptance limit) < Lower Acceptance Limit	J-	UJ
Lower Acceptance limit ≤ %R ≤ Upper Acceptance Limit	No qualification	No qualification
%R > Upper Acceptance Limit	J+	No qualification

List the percent recoveries (%Rs) which do not meet the criteria for DMCs (surrogate) recovery.

Matrix: Groundwater

SAMPLE ID	SURROGATE COMPOUND	ACTION
<u>DMCs meet the required criteria in all samples analyzed. Non-deuterated surrogates added to the samples and were within laboratory recovery limits.</u>		

**Note:**

## DATA REVIEW WORKSHEETS

**Table 8. Semivolatile DMCs and the Associated Target Analytes**

<b>1,4-Dioxane-d<sub>8</sub> (DMC-1)</b>	<b>Phenol-d<sub>5</sub> (DMC-2)</b>	<b>Bis(2-Chloroethyl) ether-d<sub>8</sub> (DMC-3)</b>
1,4-Dioxane	Benzaldehyde Phenol	Bis(2-chloroethyl) ether 2,2'-Oxybis(1-chloropropane) Bis(2-chloroethoxy)methane
<b>2-Chlorophenol-d<sub>4</sub> (DMC-4)</b>	<b>4-Methylphenol-d<sub>8</sub> (DMC-5)</b>	<b>4-Chloroaniline-d<sub>4</sub> (DMC-6)</b>
2-Chlorophenol	2-Methylphenol 3-Methylphenol 4-Methylphenol 2,4-Dimethylphenol	4-Chloroaniline Hexachlorocyclopentadiene Dichlorobenzidine
<b>Nitrobenzene-d<sub>5</sub> (DMC-7)</b>	<b>2-Nitrophenol-d<sub>4</sub> (DMC-8)</b>	<b>2,4-Dichlorophenol-d<sub>3</sub> (DMC-9)</b>
Acetophenone N-Nitroso-di-n-propylamine Hexachloroethane Nitrobenzene 2,6-Dinitrotoluene 2,4-Dinitrotoluene N-Nitrosodiphenylamine	Isophorone 2-Nitrophenol	2,4-Dichlorophenol Hexachlorobutadiene Hexachlorocyclopentadiene 4-Chloro-3-methylphenol 2,4,6-Trichlorophenol 2,4,5-Trichlorophenol 1,2,4,5-Tetrachlorobenzene *Pentachlorophenol 2,3,4,6-Tetrachlorophenol
<b>Dimethylphthalate-d<sub>6</sub> (DMC-10)</b>	<b>Acenaphthylene-d<sub>8</sub> (DMC-11)</b>	<b>4-Nitrophenol-d<sub>4</sub> (DMC-12)</b>
Caprolactam 1,1'-Biphenyl Dimethylphthalate Diethylphthalate Di-n-butylphthalate Butylbenzylphthalate Bis(2-ethylhexyl) phthalate Di-n-octylphthalate	*Naphthalene *2-Methylnaphthalene 2-Chloronaphthalene *Acenaphthylene *Acenaphthene	2-Nitroaniline 3-Nitroaniline 2,4-Dinitrophenol 4-Nitrophenol 4-Nitroaniline

# DATA REVIEW WORKSHEETS

<b>Fluorene-d<sub>10</sub> (DMC-13)</b>	<b>4,6-Dinitro-2-methylphenol-d<sub>2</sub> (DMC-14)</b>	<b>Anthracene-d<sub>10</sub> (DMC-15)</b>
Dibenzofuran *Fluorene 4-Chlorophenyl-phenylether 4-Bromophenyl-phenylether Carbazole	4,6-Dinitro-2-methylphenol	Hexachlorobenzene Atrazine *Phenanthrene *Anthracene
<b>Pyrene-d<sub>10</sub> (DMC-16)</b>	<b>Benzo(a)pyrene-d<sub>12</sub> (DMC-17)</b>	
*Fluoranthene *Pyrene *Benzo(a)anthracene *Chrysene	3,3'-Dichlorobenzidine *Benzo(b)fluoranthene *Benzo(k)fluoranthene *Benzo(a)pyrene *Indeno(1,2,3-cd)pyrene *Dibenzo(a,h)anthracene *Benzo(g,h,i)perylene	

\*Included in optional Target Analyte List (TAL) of PAHs and PCP only.

**Table 9. Semivolatile SIM DMCs and the Associated Target Analytes**

<b>Fluoranthene-d<sub>10</sub> (DMC-1)</b>	<b>2-Methylnaphthalene-d<sub>10</sub> (DMC-2)</b>
Fluoranthene	Naphthalene
Pyrene	2-Methylnaphthalene
Benzo(a)anthracene	Acenaphthylene
Chrysene	Acenaphthene
Benzo(b)fluoranthene	Fluorene
Benzo(k)fluoranthene	Pentachlorophenol
Benzo(a)pyrene	Phenanthrene
Indeno(1,2,3-cd)pyrene	Anthracene
Dibenzo(a,h)anthracene	
Benzo(g,h,i)perylene	

## DATA REVIEW WORKSHEETS

All criteria were met \_\_\_\_\_  
 Criteria were not met \_\_\_\_\_  
 and/or see below   X  

### VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples. If any % R in the MS or MSD falls outside the designated range, the reviewer should determine if there are matrix effects, i.e. LCS data are within the QC limits but MS/MSD data are outside QC limit.

#### 1. MS/MSD Recoveries and Precision Criteria

The laboratory should use one MS and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If target analytes are not expected, MS/MSD should be analyzed.

**NOTES:** Data for MS and MSDs will not be present unless requested by the Region.  
 Notify the Contract Laboratory COR if a field or trip blank was used for the MS and MSD.

For a Matrix Spike that does not meet criteria, apply the action to only the field sample used to prepare the Matrix Spike sample. If it is clearly stated in the data validation materials that the samples were taken through incremental sampling or some other method guaranteeing the homogeneity of the sample group, then the entire sample group may be qualified.

List the %Rs, RPD of the compounds which do not meet the criteria.

Sample ID: <u>FA41854-2</u>	Matrix/Level: <u>Groundwater</u>
Sample ID: <u>JC41854-2 (SIM)</u>	Matrix/Level: <u>Groundwater</u>
Sample ID: <u>JC41854-2 (SIM)</u>	Matrix/Level: <u>Groundwater</u>

The QC reported here applies to the following samples:  
 FA41854-1, FA41854-2

Method: SW846 8270D

Compound	FA41854-2 ug/l	Q	Spike ug/l	MS ug/l	MS %	Spike ug/l	MSD ug/l	MSD %	RPD	Limits Rec/RPD
bis(2-Ethylhexyl)- phthalate	ND		96.2	142	148*	96.2	133	138*	7	61-117/23

\* - outside laboratory control limits

**Note:** MS/MSD % recovery and RPD within laboratory control limits except for the cases described in this document. bis(2-ethylhexyl)phthalate not detected in sample batch, no qualification performed.

Two separate spike samples were analyzed in the SIM mode; one for 1,4-dioxane and one for naphthalene and benzo(a)anthracene.

## DATA REVIEW WORKSHEETS

- \* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- \* If QC limits are not available, use limits of 70 – 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

MS/MSD criteria apply only to the unspiked sample, its dilutions, and the associated MS/MSD samples:

If the % R for the affected compounds were < LL (or 70 %), qualify positive results (J) and nondetects (UJ).

If the % R for the affected compounds were > UL (or 130 %), only qualify positive results (J).

If 25 % or more of all MS/MSD %R were < LL (or 70 %) or if two or more MS/MSD %Rs were < 10%, qualify all positive results (J) and reject nondetects (R).

A separate worksheet should be used for each MS/MSD pair.

## DATA REVIEW WORKSHEETS

All criteria were met ☒   
 Criteria were not met   
 and/or see below \_\_\_\_\_

### INTERNAL STANDARD PERFORMANCE

The assessment of the internal standard (IS) parameter is used to assist the data reviewer in determining the condition of the analytical instrumentation.

List the internal standard area of samples which do not meet the criteria.

DATE	SAMPLE ID	IS OUT	IS AREA	ACCEPTABLE RANGE	ACTION
Internal area meets the required criteria for batch samples corresponding to this data package.					

#### Action:

1. If an internal standard area count for a sample or blank is greater than 213.0% of the area for the associated standard (opening CCV or mid-point standard from initial calibration) (see Table 10 below):
  - a. Qualify detects for compounds quantitated using that internal standard as estimated low (J-).
  - b. Do not qualify non-detected associated compounds.
2. If an internal standard area count for a sample or blank is less than 20.0% of the area for the associated standard (opening CCV or mid-point standard from initial calibration):
  - a. Qualify detects for compounds quantitated using that internal standard as estimated high (J+).
  - b. Qualify non-detected associated compounds as unusable (R).
3. If an internal standard area count for a sample or blank is greater than or equal to 50.0%, and less than or equal to 213% of the area for the associated standard opening CCV or mid-point standard from initial calibration, no qualification of the data is necessary.
4. If an internal standard RT varies by more than 10.0 seconds: Examine the chromatographic profile for that sample to determine if any false positives or negatives exist. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for that sample fraction. Detects should not need to be qualified as unusable (R) if the mass spectral criteria are met.
5. If an internal standard RT varies by less than or equal to 10.0 seconds, no qualification of the data is necessary.

## DATA REVIEW WORKSHEETS

**Note:** Inform the Contract Laboratory Program Project Officer (CLP PO) if the internal standard performance criteria are grossly exceeded. Note in the Data Review Narrative potential effects on the data resulting from unacceptable internal standard performance.

State in the Data Review Narrative if the required internal standard compounds are not added to a sample or blank or if the required internal standard compound is not analyzed at the specified concentration.

Actions:

**Table 10. Internal Standard Actions for Semivolatile Analysis**

Criteria	Action	
	Detect	Non-detect
Area response < 20% of the opening CCV or mid-point standard CS3 from ICAL	J+	R
20% ≤ Area response < 50% of the opening CCV or mid-point standard CS3 from ICAL	J+	UJ
50% ≤ Area response ≤ 200% of the opening CCV or mid-point standard CS3 from ICAL	No qualification	No qualification
Area response > 200% of the opening CCV or mid-point standard CS3 from ICAL	J-	No qualification
RT shift between sample/blank and opening CCV or mid-point standard CS3 from ICAL > 10.0 seconds	R	R
RT shift between sample/blank and opening CCV or mid-point standard CS3 from ICAL < 10.0 seconds	No qualification	No qualification

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met  
 and/or see below           

### TARGET COMPOUND IDENTIFICATION

#### Criteria:

Is the Relative Retention Times (RRTs) of reported compounds within  $\pm 0.06$  RRT units of the standard RRT [opening Continuing Calibration Verification (CCV) or mid-point standard from the initial calibration].  
**Yes? or No?**

List compounds not meeting the criteria described above:

Sample ID	Compounds	Actions
=====	=====	=====
_____	_____	_____
_____	_____	_____
_____	_____	_____

Mass spectra of the sample compound and a current laboratory-generated standard [i.e., the mass spectrum from the associated calibration standard (opening CCV or mid-point standard from initial calibration)] must match according to the following criteria:

- All ions present in the standard mass spectrum at a relative intensity greater than 10% must be present in the sample spectrum.
- The relative intensities of these ions must agree within  $\pm 20\%$  between the standard and sample spectra (e.g., for an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30-70%).
- Ions present at greater than 10% in the sample mass spectrum, but not present in the standard spectrum, must be evaluated by a reviewer experienced in mass spectral interpretation.

List compounds not meeting the criteria described above:

Sample ID	Compounds	Actions
=====	=====	=====
_____	_____	_____
_____	_____	_____
_____	_____	_____

Identified compounds meet the required criteria



## DATA REVIEW WORKSHEETS

### Action:

1. The application of qualitative criteria for GC/MS analysis of target compounds requires professional judgment. It is up to the reviewer's discretion to obtain additional information from the laboratory. If it is determined that incorrect identifications were made, qualify all such data as unusable (R).
2. Use professional judgment to qualify the data if it is determined that cross-contamination has occurred.
3. Note in the Data Review Narrative any changes made to the reported compounds or concerns regarding target compound identifications. Note, for Contract Laboratory COR action, the necessity for numerous or significant changes.

### TENTATIVELY IDENTIFIED COMPOUNDS (TICS)

NOTE: Tentatively identified compounds should only be evaluated when requested by a party from outside of the Hazardous Waste Support Section (HWSS).

#### List TICs

Sample ID	Compound	Sample ID	Compound
=====			

### Action:

1. Qualify all TIC results for which there is presumptive evidence of a match (e.g. greater than or equal to 85% match) as tentatively identified (NJ), with approximated concentrations. TICs labeled "unknown" are qualified as estimated (J).
2. General actions related to the review of TIC results are as follows:
  - a. If it is determined that a tentative identification of a non-target compound is unacceptable, change the tentative identification to "unknown" or another appropriate identification, and qualify the result as estimated (J).
  - b. If all contractually-required peaks were not library searched and quantitated, the Region's designated representative may request these data from the laboratory.
3. In deciding whether a library search result for a TIC represents a reasonable identification, use professional judgment. If there is more than one possible match, report the result as "either compound X or compound Y". If there is a lack of isomer specificity, change the TIC result to a nonspecific isomer result (e.g., 1,3,5-trimethyl benzene to trimethyl benzene isomer) or to a compound class (e.g., 2-methyl, 3-ethyl benzene to a substituted aromatic compound).
4. The reviewer may elect to report all similar compounds as a total (e.g., all alkanes may be summarized and reported as total hydrocarbons).

## DATA REVIEW WORKSHEETS

5. Target compounds from other fractions and suspected laboratory contaminants should be marked as "non-reportable".
6. Other Case factors may influence TIC judgments. If a sample TIC match is poor, but other samples have a TIC with a valid library match, similar RRT, and the same ions, infer identification information from the other sample TIC results.
7. Note in the Data Review Narrative any changes made to the reported data or any concerns regarding TIC identifications.
8. Note, for Contract Laboratory COR action, failure to properly evaluate and report TICs

## DATA REVIEW WORKSHEETS

All criteria were met X  
 Criteria were not met  
 and/or see below \_\_\_\_\_

### SAMPLE QUANTITATION AND REPORTED CONTRACT REQUIRED QUANTITATION LIMITS (CRQLS)

#### Action:

1. When a sample is analyzed at more than one dilution, the lower CRQL are used unless a QC exceedance dictates the use of higher CRQLs from the diluted sample. Samples reported with an "E" qualifier should be reported from the diluted sample.
2. If any discrepancies are found, the Region's designated representative may contact the laboratory to obtain additional information that could resolve any differences. If a discrepancy remains unresolved, the reviewer must use professional judgment to decide which value is the most accurate. Under these circumstances, the reviewer may determine that qualification of data is warranted. Note in the Data Review Narrative a description of the reasons for data qualification and the qualification that is applied to the data.
3. For non-aqueous samples, if the solids is less than 10.0%, use professional judgment for both detects and non-detects. If the percent solid for a soil sample is greater than or equal to 10.0% and less than 30.0%, use professional judgment to qualify detects and non-detects. If the percent solid for a soil sample is greater than or equal to 30.0%, detects and non-detects should not be qualified (see Table 11).
4. Note, for Contract Laboratory COR action, numerous or significant failures to accurately quantify the target compounds or to properly evaluate and adjust CRQLs.
5. Results between MDL and CRQL should be qualified as estimated "J".
6. Results < MDL should be reported at the CRQL and qualified "U". MDLs themselves should not be reported.

**Table 11. Percent Solids Actions for Semivolatile Analysis for Non-Aqueous Samples**

Criteria	Action	
	Detects	Non-detects
%Solids < 10.0%	Use professional judgment	Use professional judgment
10.0% ≤ %Solids ≤ 30.0%	Use professional judgment	Use professional judgment
%Solids > 30.0%	No qualification	No qualification

### SAMPLE QUANTITATION

The sample quantitation evaluation is to verify laboratory quantitation results. In the space below, please show a minimum of one sample calculation:

Sample ID: FA41854-1 Analyte: 1,4-dixane RF: 0.599

$$\begin{aligned}
 [ ] &= (3510)(4.0)/(31040)(0.599) \\
 &= 0.76 \text{ ppm} \quad \text{Ok}
 \end{aligned}$$

• • • •

## QUANTITATION LIMITS

A. Dilution performed

[illegible]

## DATA REVIEW WORKSHEETS

All criteria were met \_\_\_\_\_  
 Criteria were not met \_\_\_\_\_  
 and/or see below \_\_\_\_\_N/A\_\_\_\_\_

### FIELD DUPLICATE PRECISION

Sample IDs: \_\_\_\_\_ - \_\_\_\_\_

Matrix: \_\_\_\_\_

Field duplicates samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

The project QAPP should be reviewed for project-specific information.

Suggested criteria: if large RPD (> 50 %) is observed, confirm identification of the samples and note differences. If both samples and duplicate are <5 SQL, the RPD criteria is doubled.

COMPOUND	SQL ug/L	SAMPLE CONC. (ug/l)	DUPLICATE CONC. (ug/l)	RPD	ACTION
No field/laboratory duplicate analyzed as part of this data package. MS/MSD % recovery RPD used to assess precision. RPD within the required guidance document criteria < 50 % for detected target analytes above 5 SQL.					

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met  
 and/or see below \_\_\_\_\_

### OTHER ISSUES

#### A. System Performance

List samples qualified based on the degradation of system performance during sample analysis:

Sample ID	Comments	Actions
=====	=====	=====
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

Action:

Use professional judgment to qualify the data if it is determined that system performance has degraded during sample analyses. Inform the Contract Laboratory Program COR any action as a result of degradation of system performance which significantly affected the data.

#### B. Overall Assessment of Data

List samples qualified based on other issues:

Sample ID	Comments	Actions
=====	=====	=====
_No_other_issues_that_required_the_need_to_qualify_the_data._Results_are_valid_and_can_be_used_for_decision_purposes._Other_discrepancies_are_shown_below._____		
_____	_____	_____
_____	_____	_____

**Note:** bis(2-Ethylhexyl)phthalate recover high in Blank Spike. No action taken, professional judgment; bis(2-Ethylhexyl)phthalate not detected in sample batch.

Action:

1. Use professional judgment to determine if there is any need to qualify data which were not qualified based on the Quality Control (QC) criteria previously discussed.
2. Write a brief narrative to give the user an indication of the analytical limitations of the data. Inform the Contract Laboratory COR the action, any inconsistency of the data with the Sample Delivery Group (SDG) Narrative. If sufficient information on the intended use and required quality of the data is available, the reviewer should include their assessment of the usability of the data within the given context. This may be used as part of a formal Data Quality Assessment (DQA).

## DATA REVIEW WORKSHEETS

3. Sometimes, due to dilutions, re-analysis or SIM/Scan runs are being performed, there will be multiple results for a single analyte from a single sample. The following criteria and professional judgment are used to determine which result should be reported:
  - The analysis with the lower CRQL
  - The analysis with the better QC results
  - The analysis with the higher results

## EXECUTIVE NARRATIVE

SDG No: **FA41854** Laboratory: **Accutest, Orlando**

Analysis: **MADEP VPH** Number of Samples: **2**

Location: **BMSMC, Humacao, PR**

SUMMARY: Two (2) samples were analyzed for Volatiles TPHC Ranges by method MADEP VPH. Samples were validated following the METHOD FOR THE DETERMINATION OF VOLATILE PETROLEUM HYDROCARBONS (VPH) quality control criteria, Massachusetts Department of Environmental Protection, Revision 1.1 (2004). Also the general validation guidelines promulgated by the USEPA Hazardous Wastes Support Section. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

Results are valid and can be used for decision making purposes.

Critical issues: **None**

Major: **None**

Minor: **None**

Critical findings: **None**

Major findings: **None**

Minor findings: **1. MS/MSD % recovery outside laboratory control limits for C9-C10 Aromatics (Unadj).  
NO action taken, MS/MSD samples were from another job.**

COMMENTS: **Results are valid and can be used for decision making purposes.**

Reviewers Name: **Rafael Infante**  
**Chemist License 1888**

Signature:



Date: **April 14, 2017**



ORGANIC DATA SAMPLE SUMMARY

Sample ID: FA41854-1

Sample location: BMSMC, Humacao, PR

Sampling date: 3/6/2017

Matrix: Groundwater

METHOD: MADEP VPH

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
C9 - C10 Aromatics (Unadj.)	100	ug/L	1.0	-	U	Yes

Sample ID: FA41854-2

Sample location: BMSMC, Humacao, PR

Sampling date: 3/6/2017

Matrix: Groundwater

METHOD: MADEP VPH

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
C9 - C10 Aromatics (Unadj.)	100	ug/L	1.0	-	U	Yes

# DATA REVIEW WORKSHEETS

Type of validation Full: ☒ Limited: \_\_\_\_\_  
 Project Number: FA41854  
 Date: 03/06/2017  
 Shipping date: 03/07/2017  
 EPA Region: 2

## REVIEW OF VOLATILE PETROLEUM HYDROCARBON (VPHs) PACKAGE

The following guidelines for evaluating volatile organics were created to delineate required validation actions. This document will assist the reviewer in using professional judgment to make more informed decision and in better serving the needs of the data users. The sample results were assessed according to the data validation guidance documents in the following order of precedence METHOD FOR THE DETERMINATION OF VOLATILE PETROLEUM HYDROCARBONS (VPH), Massachusetts Department of Environmental Protection, Revision 1.1 (2004). Also the general validation guidelines promulgated by the USEPA Hazardous Wastes Support Section. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

The hardcopied (laboratory name) Accutest Laboratories - Orlando data package received has been reviewed and the quality control and performance data summarized. The data review for VOCs included:


Lab. Project/SDG No.: FA41854 Sample matrix: Groundwater  
 No. of Samples: 2  
 Field blank No.: -  
 Equipment blank No.: -  
 Trip blank No.: -  
 Field duplicate No.: -

<input checked="" type="checkbox"/> Data Completeness	<input checked="" type="checkbox"/> Laboratory Control Spikes
<input checked="" type="checkbox"/> Holding Times	<input checked="" type="checkbox"/> Field Duplicates
<input type="checkbox"/> GC/MS Tuning	<input checked="" type="checkbox"/> Calibrations
<input type="checkbox"/> Internal Standard Performance	<input checked="" type="checkbox"/> Compound Identifications
<input checked="" type="checkbox"/> Blanks	<input checked="" type="checkbox"/> Compound Quantitation
<input checked="" type="checkbox"/> Surrogate Recoveries	<input checked="" type="checkbox"/> Quantitation Limits
<input checked="" type="checkbox"/> Matrix Spike/Matrix Spike Duplicate	

Overall Comments: Volatiles by GC by Method MADEP\_VPH\_REV\_1.1\_(C9- C10 Aromatics (Unadj.))

### Definition of Qualifiers:

J- Estimated results  
 U- Compound not detected  
 R- Rejected data  
 UJ- Estimated nondetect

Reviewer:   
 Date: April 14, 2017

## DATA REVIEW WORKSHEETS

All criteria were met   x    
Criteria were not met and/or see below           

### I. DATA COMPLETNESS

#### A. Data Package:

MISSING INFORMATION

DATE LAB. CONTACTED

DATE RECEIVED


#### B. Other

Discrepancies:


## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met and/or see below       

### HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of extraction, and subsequently from the time of extraction to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE EXTRACTED	DATE ANALYZED	ACTION
Samples analyzed within method recommended holding time. Sample preservation within the required criteria.				

### Criteria

#### Preservation:

Samples analyzed with ambient purge temperature: Samples must be acidified to a pH of 2.0 or less at the time of collection.

Samples analyzed with heated purge temperature: Samples must be treated to a pH of 11.0 or greater at the time of collection.

Methanol preservation of soil/sediment samples is mandatory. Methanol (purge-and-trap grade) must be added to the sample vial before or immediately after sample collection. In lieu of the in-field preservation of samples with methanol, soil samples may be obtained in specially-designed air tight sampling devices, provided that the samples are extruded and preserved in methanol within 48 hours of collection.

#### Holding times:

Aqueous samples using ambient or heated purge - analyze within 14 days.

Soil/sediment samples - analysis within 28 days.

Cooler temperature (Criteria:  $4 \pm 2$  °C):   3.0/3.2   °C

#### Actions: Qualify positive results/non-detects as follows:

If holding times are exceeded, estimate positive results (J) and nondetects (UJ).

If holding times are grossly exceeded, use professional judgment to qualify data. The data reviewer may choose to estimate positive results (J) and rejects nondetects (R).

If samples were not at the proper temperature ( $> 10^{\circ}\text{C}$ ) or improperly preserved, use professional judgment to qualify the results.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met and/or see below           

### CALIBRATIONS VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration:   03/06/17;\_03/17/17  

Dates of initial calibration verification:   03/06/17;\_03/17/1  

Instrument ID numbers:                      VOA10                     

Matrix/Level:                      AQUEOUS/MEDIUM                     

DATE	LAB FILE ID#	ANALYTE	CRITERIA OUT RFs, %RSD, %D, r	SAMPLES AFFECTED
Initial and initial calibration verification meet method specific requirements				

#### Criteria- ICAL

- Five point calibration curve.
- The percent relative standard deviation (%RSD) of the calibration factor must be equal to or less than 25% over the working range for the analyte of interest. When this condition is met, linearity through the origin may be assumed, and the average calibration factor is used in lieu of a calibration curve.
- A collective calibration factor must also be established for each hydrocarbon range of interest. Calculate the collective CFs for C5-C8 Aliphatic Hydrocarbons and C9-C12 Aliphatic Hydrocarbons using the FID chromatogram. Calculate the collective CF for the C9-C10 Aromatic Hydrocarbons using the PID chromatogram. Tabulate the summation of the peak areas of all components in that fraction against the total concentration injected. The %RSD of the calibration factor must be equal to or less than 25% over the working range for the hydrocarbon range of interest.

#### Criteria- CCAL

- At a minimum, the working calibration factor must be verified on each working day, after every 20 samples, and at the end of the analytical sequence by the injection of a mid-level continuing calibration standard to verify instrument performance and linearity.
- If the percent difference (%D) for any analyte varies from the predicted response by more than  $\pm 25\%$ , a new five-point calibration must be performed for that analyte. Greater percent differences are permissible for n-nonane. If the %D for n-nonane is greater than 30, note the nonconformance in the case narrative. It should be noted that the %Ds are calculated when CFs are used for the initial calibration and

## DATA REVIEW WORKSHEETS

percent drifts are calculated when calibration curves using linear regression are used for the initial calibration.

### Actions:

If %RSD > 25% for target compounds or a correlation coefficient < 0.99, estimate positive results (J) and use professional judgment to qualify nondetects.

If % D > 25% (> 30 for nonane), estimate positive results (J) and nondetects (UJ).

### CALIBRATIONS VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration: 03/06/17  
 Dates of continuing calibration verification: 03/09/17; 03/10/17; 03/15/17  
 Dates of final calibration verification: 03/10/17  
 Instrument ID numbers: VOA10  
 Matrix/Level: AQUEOUS/MEDIUM

Date of initial calibration: 03/17/17  
 Dates of continuing calibration verification: 03/17/17  
 Dates of final calibration verification: 03/17/17  
 Instrument ID numbers: VOA10  
 Matrix/Level: AQUEOUS/MEDIUM

DATE	LAB FILE ID#	ANALYTE	CRITERIA OUT RFs, %RSD, %D, r	SAMPLES AFFECTED

**Note:** Continuing and final calibration verification meets method specific requirements.

A separate worksheet should be filled for each initial curve

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met and/or see below           

### V A. BLANK ANALYSIS RESULTS (Sections 1 & 2)

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data. A Laboratory Method Blank must be run after samples suspected of being highly contaminated to determine if sample carryover has occurred.

List the contamination in the blanks below. High and low levels blanks must be treated separately.

Laboratory blanks

DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
------------------	--------	------------------	----------	------------------------

\_METHOD\_BLANKS\_MEET\_THE\_METHOD\_SPECIFIC\_CRITERIA\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

**Note:**

Field/Trip/Equipment

A methanol trip blank or acidified reagent water trip blank **should** continually accompany each soil/sediment sample or water sample batch, respectively, during sampling, storage, and analysis.

DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
------------------	--------	------------------	----------	------------------------

\_NO\_TRIP/FIELD/EQUIPMENT\_BLANKS\_ASSOCIATED\_WITH\_THIS\_DATA\_PACKAGE  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

**Note:**

## DATA REVIEW WORKSHEETS

### V B. BLANK ANALYSIS RESULTS (Section 3)

#### Blank Actions

The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. Peaks must not be detected above the Reporting Limit within the retention time window of any analyte of interest. The hydrocarbon ranges must not be detected at a concentration greater than 10% of the most stringent MCP cleanup standard. Specific actions area as follows:

If the concentration is  $<$  sample quantitation limit (SQL) and  $<$  AL, report the compound as not detected (U) at the SQL.

If the concentration is  $\geq$  SQL but  $<$  AL, report the compound as not detected (U) at the reported concentration.

If the concentration is  $>$  AL, report the concentration unqualified.



## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met and/or see below           

### SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery.

Matrix: solid/aqueous

SAMPLE ID	SURROGATE COMPOUND	ACTION
	BFB	

  SURROGATE STANDARD RECOVERIES WITHIN LABORATORY CONTROL  
  LIMITS. SURROGATE RECOVERIES WERE CORRECTED FOR ACTUAL SPIKE  
  AMOUNT.  

QC Limits\* (Aqueous)

       LL   to   UL          70   to  130         to               to       

QC Limits\* (Solid)

       LL   to   UL          70   to  130         to               to       

It is recommended that surrogate standard recoveries be monitored and documented on a continuing basis. At a minimum, when surrogate recovery from a sample, blank, or QC sample is less than 70% or more than 130%, check calculations to locate possible errors, check the fortifying standard solution for degradation, and check changes in instrument performance.

If the cause cannot be determined, reanalyze the sample unless one of the following exceptions applies:

- (1) Obvious interference is present on the chromatogram (e.g., unresolved complex mixture);
- (2) Percent moisture of associated soil/sediment sample is >25% and surrogate recovery is >10%; or
- (3) The surrogate exhibits high recovery and associated target analytes or hydrocarbon ranges are not detected in sample.

If a sample with a surrogate recovery outside of the acceptable range is not reanalyzed based on any of these aforementioned exceptions, this information must be noted on the data report form and discussed in the Executive Report. Analysis of the sample on dilution may diminish matrix-related surrogate recovery problems. This approach can be used as long as the reporting limits to evaluate applicable MCP standards can still be achieved with the dilution. If not, reanalysis without dilution must be performed.

## DATA REVIEW WORKSHEETS

All criteria were met \_\_\_\_\_  
 Criteria were not met and/or see below X

### VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples.

At the request of the data user, and in consideration of sample matrices and data quality objectives, matrix spikes and matrix duplicates may be analyzed with every batch of 20 samples or less per matrix.

- **Matrix duplicate** - Matrix duplicates are prepared by analyzing one sample in duplicate. The purpose of the matrix duplicates is to determine the homogeneity of the sample matrix as well as analytical precision. The RPD of detected results in the matrix duplicate samples must not exceed 50 when the results are greater than 5x the reporting limit.
- The desired spiking level is 50% of the highest calibration standard. However, the total concentration in the MS (including the MS and native concentration in the unspiked sample) should not exceed 75% of the highest calibration standard in order for a proper evaluation to be performed. The purpose of the matrix spike is to determine whether the sample matrix contributes bias to the analytical results. The corrected concentrations of each analyte within the matrix spiking solution must be within 70 - 130% of the true value. Lower recoveries of n-nonane are permissible (if included in the calibration of the C9-C12 aliphatic range), but must be noted in the narrative if <30%.

#### MS/MSD Recoveries and Precision Criteria

Sample ID: FA41752-2\_MS/MSD  
 Sample ID: FA41811-2\_MS/MSD

Matrix/Level: Groundwater  
 Matrix/Level: Groundwater

List the %Rs, RPD of the compounds which do not meet the QC criteria.

The QC reported here applies to the following samples:  
**FA41854-1**

Method: **MADEP VPH REV 1.1**

Compound	FA41752-2 ug/l	Q	Spike ug/l	MS ug/l	MS %	Spike ug/l	MSD ug/l	MSD %	RPD	Limits Rec/RPD
C9- C10 Aromatics (Unadj.)	ND		240	86.1	36*	240	86.0	36*	0	70-130/50

The QC reported here applies to the following samples:  
**FA41854-2**

Method: **MADEP VPH REV 1.1**

Compound	FA41811-2 ug/l	Q	Spike ug/l	MS ug/l	MS %	Spike ug/l	MSD ug/l	MSD %	RPD	Limits Rec/RPD
C9- C10 Aromatics (Unadj.)	ND		240	89.6	37*	240	87.6	37*	2	70-130/50

\* Outside laboratory control limits.

## DATA REVIEW WORKSHEETS

**Note:** MS/MSD % recovery and RPD within laboratory control limits except for the cases described in this document. No action taken, MS/MSD samples were from another job.

No action is taken on MS/MSD results alone to qualify the entire case. However, used informed professional judgment, the data reviewer may use the MS/MSD results in conjunction with other QC criteria and determine the need for some qualification of the data. In those instances where it can be determined that the results of the MS/MSD affect only the sample spiked, the qualification should be limited to this sample alone. However, it may be determined through the MS/MSD results that the laboratory is having a systematic problem in the analysis of one or more analytes, which affects the associated samples.

### 2. MS/MSD – Unspiked Compounds

List the concentrations of the unspiked compounds and determine the % RSDs of these compounds in the unspiked sample, matrix spike, and matrix spike duplicate.

COMPOUND	CONCENTRATION		MSD	%RPD	ACTION
	SAMPLE	MS			

Criteria: None specified, use %RSD  $\leq$  50 as professional judgment.

Actions:

If the % RSD > 50, qualify the results in the spiked sample as estimate (J).

If the % RSD is not calculable (NC) due to nondetect value in the sample, MS, and/or MSD, use professional judgment to qualify sample data.

A separate worksheet should be used for each MS/MSD pair.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met and/or see below           

### VIII. LABORATORY CONTROL SAMPLE (LCS/LCSD) ANALYSIS

This data is generated to determine accuracy of the analytical method for various matrices.

#### 1. LCS Recoveries Criteria

List the %R of compounds which do not meet the criteria

LCS ID	COMPOUND	% R	QC LIMIT	ACTION
--------	----------	-----	----------	--------

  LCS/LCSD\_RECOVERY\_WITHIN\_LABORATORY\_CONTROL\_LIMITS.          


#### Criteria:

- \* Refer to QAPP for specific criteria.
- \* The spike recovery must be between 70% and 130%. Lower recoveries of n-nonane are permissible (if included in the calibration of the C9-C12 aliphatic range). If the recovery of n-nonane is <30%, note the nonconformance in the executive narrative.

#### Actions:

Actions on LCS recovery should be based on both the number of compounds that are outside the %R criteria and the magnitude of the exceedance of the criteria.

If the %R of the analyte is > UL, qualify all positive results (j) for the affected analyte in the associated samples and accept nondetects.

If the %R of the analyte is < LL, qualify all positive results (j) and reject (R) nondetects for the affected analyte in the associated samples.

If more than half the compounds in the LCS are not within the required recovery criteria, qualify all positive results as (J) and reject nondetects (R) for all target analyte(s) in the associated samples.

#### 2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix (1 per 20 samples per matrix)? Yes or No.

If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected. Discuss the actions below:

## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met and/or see below           

### IX. FIELD/LABORATORY DUPLICATE PRECISION

Sample IDs:   FA42015-5/FA42015-5DUP  

Matrix:   Aqueous  

Field/laboratory duplicates samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which measures only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION
Field/laboratory duplicate analyzed with this data package. RPD within laboratory and validation guidance document criteria ( $\pm 50\%$ ) for analytes detected above reporting limits.					

#### Criteria:

The project QAPP should be reviewed for project-specific information.  
RPD  $\pm 30\%$  for aqueous samples, RPD  $\pm 50\%$  for solid samples if results are  $\geq$  SQL.  
If both samples and duplicate are  $<5$  SQL, the RPD criteria is doubled.

SQL = soil quantitation limit

#### Actions:

If both the sample and the duplicate results are nondetects (ND), the RPD is not calculable (NC). No action is needed.

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria.

If one sample result is not detected and the other is  $\geq 5x$  the SQL qualify (J/UJ).

**Note:** If SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is  $< 5x$  the SQL, use professional judgment to determine if qualification is appropriate.

All criteria were met   X    
Criteria were not met and/or see below           

## XI. COMPOUND IDENTIFICATION

The compound identification evaluation is to verify that the laboratory correctly identified target analytes as well as tentatively identified compounds (TICs).

1. Verify that the target analytes were within the retention time windows.
  - Retention time windows must be re-established for each Target VPH Analyte each time a new GC column is installed, and must be verified and/or adjusted on a daily basis.
  - Coelution of the m- and p- xylene isomers is permissible.
  - All surrogates must be adequately resolved from individual Target Analytes included in the VPH Component Standard.
  - For the purposes of this method, adequate resolution is assumed to be achieved if the height of the valley between two peaks is less than 25% of the average height of the two peaks.
  - The n-pentane (C5) and MtBE peaks must be adequately resolved from any solvent front that may be present on the FID and PID chromatograms, respectively.

**Note:** Target analytes were within the retention time window.

2. If target analytes and/or TICs were not correctly identified, request that the laboratory resubmit the corrected data.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met and/or see below           

### XII. QUANTITATION LIMITS AND SAMPLE RESULTS

The sample quantitation evaluation is to verify laboratory quantitation results.

1. In the space below, please show a minimum of one sample calculation:

FID

Computer printout

PID

Computer printout

2. If requested, verify that the results were above the laboratory method detection limit (MDLs).
3. If dilutions performed, were the SQLs elevated accordingly by the laboratory? List the affected samples and dilution factor in the table below.

SAMPLE ID	DILUTION FACTOR	REASON FOR DILUTION

If dilution was not performed and the results were above the concentration range, estimate results (J) for the affected compounds. List the affected samples/compounds:

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## EXECUTIVE NARRATIVE

SDG No: **FA41854** Laboratory: **Accutest, Orlando**  
Analysis: **MADEP EPH** Number of Samples: **2**

Location: **BMSMC, Humacao, PR**

**SUMMARY:** Two (2) samples were analyzed for Semivolatiles TPHC Ranges by method MADEP EPH. Samples were validated following the METHOD FOR THE DETERMINATION OF EXTRACTABLE PETROLEUM HYDROCARBONS (EPH) quality control criteria, Massachusetts Department of Environmental Protection, Revision 1.1 (2004). Also the general validation guidelines promulgated by the USEPA Hazardous Wastes Support Section. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

Results are valid and can be used for decision making purposes.

**Critical issues:** **None**  
**Major:** **None**  
**Minor:** **None**

**Critical findings:** **None**  
**Major findings:** **None**  
**Minor findings:** 1. Method blanks meet the method performance criteria except for the cases described in the Data Review Worksheet. No action taken, target analytes not detected in sample batch.

**COMMENTS:** Results are valid and can be used for decision making purposes.

**Reviewers Name:** **Rafael Infante**  
**Chemist License 1888**

**Signature:**

A handwritten signature in blue ink, reading "Rafael Infante", is written over a horizontal line.

**Date:** **April 24, 2017**



ORGANIC DATA SAMPLE SUMMARY

Sample ID: FA41854-1

Sample location: BMSMC, Humacao, PR

Sampling date: 3/6/2017

Matrix: Groundwater

METHOD: MADEP EPH

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
C11 - C22 Aromatics (Unadj.)	200	ug/L	1.0	-	U	Yes

Sample ID: FA41854-2

Sample location: BMSMC, Humacao, PR

Sampling date: 3/6/2017

Matrix: Groundwater

METHOD: MADEP EPH

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
C11 - C22 Aromatics (Unadj.)	200	ug/L	1.0	-	U	Yes

# DATA REVIEW WORKSHEETS

Type of validation      Full: ☒ Limited: \_\_\_\_\_  
 Project Number: FA41854  
 Date: 03/06/2017  
 Shipping date: 03/07/2017  
 EPA Region: 2

## REVIEW OF EXTRACTABLE PETROLEUM HYDROCARBON (EPHs) PACKAGE

The following guidelines for evaluating volatile organics were created to delineate required validation actions. This document will assist the reviewer in using professional judgment to make more informed decision and in better serving the needs of the data users. The sample results were assessed according to the data validation guidance documents in the following order of precedence METHOD FOR THE DETERMINATION OF EXTRACTABLE PETROLEUM HYDROCARBONS (EPH), Massachusetts Department of Environmental Protection, Revision 1.1 (2004). Also the general validation guidelines promulgated by the USEPA Hazardous Wastes Support Section. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

The hardcopied (laboratory name) Accutest Laboratories data package received has been reviewed and the quality control and performance data summarized. The data review for SVOCs included:

Lab. Project/SDG No.: FA41854 Sample matrix: Groundwater  
 No. of Samples: 2  
 Field blank No.: -  
 Equipment blank No.: -  
 Trip blank No.: -  
 Field duplicate No.: -

<input checked="" type="checkbox"/> Data Completeness	<input checked="" type="checkbox"/> Laboratory Control Spikes
<input checked="" type="checkbox"/> Holding Times	<input checked="" type="checkbox"/> Field Duplicates
<input type="checkbox"/> GC/MS Tuning	<input checked="" type="checkbox"/> Calibrations
<input type="checkbox"/> Internal Standard Performance	<input checked="" type="checkbox"/> Compound Identifications
<input checked="" type="checkbox"/> Blanks	<input checked="" type="checkbox"/> Compound Quantitation
<input checked="" type="checkbox"/> Surrogate Recoveries	<input checked="" type="checkbox"/> Quantitation Limits
<input checked="" type="checkbox"/> Matrix Spike/Matrix Spike Duplicate	

Overall \_\_\_\_\_ Comments: \_\_\_\_\_  
\_Extractable\_Petroleum\_Hydrocarbons\_by\_GC\_by\_Method\_MADEP\_EPH\_REV\_1.1.\_  
\_(C11\_-\_C22)\_Aromatics\_(Unadj.)

### Definition of Qualifiers:

J- Estimated results  
 U- Compound not detected  
 R- Rejected data  
 UJ- Estimated nondetect

Reviewer: Rafael Infante  
 Date: April 24, 2017

## DATA REVIEW WORKSHEETS

All criteria were met   x    
Criteria were not met and/or see below           

## I. DATA COMPLETNESS

A. Data Package:

### MISSING INFORMATION

DATE LAB. CONTACTED

DATE RECEIVED

[illegible]

B. Other

**Discrepancies:**

This image shows a single sheet of white paper with horizontal ruling lines. The lines are evenly spaced and run across the width of the page. There are no margins or other markings visible.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met and/or see below           

### HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of extraction, and subsequently from the time of extraction to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE EXTRACTED	DATE ANALYZED	ACTION
Samples extracted and analyzed within method recommended holding time				

### Criteria

#### Preservation:

Aqueous samples must be acidified to a pH of 2.0 or less at the time of collection.

Soil samples must be cooled at  $4 \pm 2$  °C immediately after collection.

#### Holding times:

Samples must be extracted within 14 days of collection, and analyzed within 40 days of extraction.

Cooler temperature (Criteria:  $4 \pm 2$  °C):   3.0/3.2   °C

#### Actions: Qualify positive results/nondetects as follows:

If holding times are exceeded, estimate positive results (J) and nondetects (UJ).

If holding times are grossly exceeded, use professional judgment to qualify data. The data reviewer may choose to estimate positive results (J) and rejects nondetects (R).

If samples were not at the proper temperature ( $> 10$ °C) or improperly preserved, use professional judgment to qualify the results.

#### **Note:**

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met and/or see below           

### CALIBRATIONS VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration: 10/29/16  
 Dates of initial calibration verification: 10/29/16  
 Instrument ID numbers: FID\_7  
 Matrix/Level: AQUEOUS/MEDIUM

Date of initial calibration: 03/15/17  
 Dates of initial calibration verification: 03/15/17  
 Instrument ID numbers: FID\_7  
 Matrix/Level: AQUEOUS/MEDIUM

DATE	LAB FILE ID#	ANALYTE	CRITERIA OUT RFs, %RSD, %D, r	SAMPLES AFFECTED
Initial and continuing calibration meet method specific requirements				

#### Criteria- ICAL

- Five point calibration curve.
- The percent relative standard deviation (%RSD) of the calibration factor must be equal to or less than 25% over the working range for the analyte of interest. When this condition is met, linearity through the origin may be assumed, and the average calibration factor is used in lieu of a calibration curve.
- A collective calibration factor must also be established for each hydrocarbon range of interest. Calculate the collective CFs for C9-C18 Aliphatic Hydrocarbons, C19-C36 Aliphatic Hydrocarbons, and C11-C22 Aromatic Hydrocarbons using the FID chromatogram. Tabulate the summation of the peak areas of all components in that fraction against the total concentration injected. The %RSD of the calibration factor must be equal to or less than 25% over the working range for the hydrocarbon range of interest.
  - The area for the surrogates must be subtracted from the area summation of the range in which they elute.
  - The areas associated with naphthalene and 2-methylnaphthalene in the aliphatic range standard must be subtracted from the uncorrected collective C9-C18 Aliphatic Hydrocarbon range area prior to calculating the CF.

## DATA REVIEW WORKSHEETS

### Criteria- CCAL

- At a minimum, the working calibration factor must be verified on each working day, after every 20 samples or every 24 hours (whichever is more frequent), and at the end of the analytical sequence by the injection of a mid-level continuing calibration standard to verify instrument performance and linearity.
- If the percent difference (%D) for any analyte varies from the predicted response by more than  $\pm 25\%$ , a new five-point calibration must be performed for that analyte. Greater percent differences are permissible for n-nonane. If the %D for n-nonane is greater than 30, note the nonconformance in the case narrative. It should be noted that the %Ds are calculated when CFs are used for the initial calibration and percent drifts are calculated when calibration curves using linear regression are used for the initial calibration.

### Actions:

If %RSD > 25% for target compounds or a correlation coefficient < 0.99, estimate positive results (J) and use professional judgment to qualify nondetects.

If % D > 25% (> 30 for nonane), estimate positive results (J) and nondetects (UJ).

### CALIBRATIONS VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration: 10/29/16  
 Dates of continuing calibration verification: 03/14/17  
 Dates of final calibration verification: 03/14/17  
 Instrument ID numbers: FID\_7  
 Matrix/Level: AQUEOUS/MEDIUM

Date of initial calibration: 03/15/17  
 Dates of continuing calibration verification: 03/16/17; 03/17/17; 03/22/17; 03/23/17  
 Dates of final calibration verification: 03/16/17; 03/17/17; 03/22/17; 03/23/17  
 Instrument ID numbers: FID\_7  
 Matrix/Level: AQUEOUS/MEDIUM

DATE	LAB FILE ID#	ANALYTE	CRITERIA OUT RFs, %RSD, %D, r	SAMPLES AFFECTED
Initial and continuing calibration meets method specific requirements.				

### Note:

A separate worksheet should be filled for each initial curve

# DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met and/or see below           

## V A. BLANK ANALYSIS RESULTS (Sections 1 & 2)

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data. A Laboratory Method Blank must be run after samples suspected of being highly contaminated to determine if sample carryover has occurred.

List the contamination in the blanks below. High and low levels blanks must be treated separately.

Laboratory blanks

DATE ANALYZED	LAB ID	LEVEL/MATRIX	COMPOUND	CONCENTRATION UNITS
---------------	--------	--------------	----------	---------------------

  METHOD BLANKS MEET THE METHOD SPECIFIC CRITERIA EXCEPT FOR    
  THE CASES DESCRIBED IN THIS DOCUMENT.  

<u>  03/14/17  </u>	<u>  OP64122-MB  </u>	<u>  AQ/LOW  </u>	<u>  C11-C22_Aromatics_(Unadj.)  </u>	<u>  80.5_ug/l  </u>
<u>  03/16/17  </u>	<u>  OP64122-MB  </u>	<u>  AQ/LOW  </u>	<u>  C11-C22_Aromatics_(Unadj.)  </u>	<u>  97.5_ug/l  </u>

**Note:** No action taken, target analytes not detected in sample batch.

Field/Trip/Equipment

DATE ANALYZED	LAB ID	LEVEL/MATRIX	COMPOUND	CONCENTRATION UNITS
---------------	--------	--------------	----------	---------------------

  NO TRIP/FIELD/EQUIPMENT BLANKS ANALYZED ASSOCIATED WITH THIS    
  DATA PACKAGE.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met and/or see below           

### V      B.      BLANK ANALYSIS RESULTS (Section 3)

#### Blank Actions

The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. Peaks must not be detected above the Reporting Limit within the retention time window of any analyte of interest. The hydrocarbon ranges must not be detected at a concentration greater than 10% of the most stringent MCP cleanup standard. Specific actions area as follows:

If the concentration is < sample quantitation limit (SQL) and < AL, report the compound as not detected (U) at the SQL.

If the concentration is  $\geq$  SQL but < AL, report the compound as not detected (U) at the reported concentration.

If the concentration is > AL, report the concentration unqualified.



## DATA REVIEW WORKSHEETS

All criteria were met X  
Criteria were not met and/or see below \_\_\_\_\_

## SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery.

**Matrix:** solid/aqueous

SAMPLE ID	SURROGATE COMPOUND				ACTION
	S1	S2	S3	S4	
_SURROGATE_STANDARDS_RECOVERIES_WITHIN_LABORATORY_CONTROL_					
_LIMITS._____					
_____					

**Note:**

S1 = o-Terphenyl 40-140%

S2 = 2-Fluorobiphenyl 40-140%

S3 = 1-Chlorooctadecane 40-140%

S4 = 2-Bromonaphthalene 40-140%

QC Limits (%)\* (Aqueous)

LL to UL    40 to 140    40 to 140    40 to 140    40 to 140

QC Limits\* (Solid)

LL to UL               to               to               to               to

It is recommended that surrogate standard recoveries be monitored and documented on a continuing basis. At a minimum, when surrogate recovery from a sample, blank, or QC sample is less than 40% or more than 140%, check calculations to locate possible errors, check the fortifying standard solution for degradation, and check changes in instrument performance.

If the cause cannot be determined, reanalyze the sample unless one of the following exceptions applies:

- (1) Obvious interference is present on the chromatogram (e.g., unresolved complex mixture);
- (2) The surrogate exhibits high recovery and associated target analytes or hydrocarbon ranges are not detected in sample.

If a sample with a surrogate recovery outside of the acceptable range is not reanalyzed based on any of these aforementioned exceptions, this information must be noted on the data report form and discussed in the Executive Report. Analysis of the sample on dilution may diminish matrix-related surrogate recovery problems. This approach can be used as long as the reporting limits to evaluate applicable MCP standards can still be achieved with the dilution. If not, reanalysis without dilution must be performed.

## DATA REVIEW WORKSHEETS

All criteria were met X  
 Criteria were not met and/or see below \_\_\_\_\_

### VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples.

At the request of the data user, and in consideration of sample matrices and data quality objectives, matrix spikes and matrix duplicates may be analyzed with every batch of 20 samples or less per matrix.

- **Matrix duplicate** - Matrix duplicates are prepared by analyzing one sample in duplicate. The purpose of the matrix duplicates is to determine the homogeneity of the sample matrix as well as analytical precision. The RPD of detected results in the matrix duplicate samples must not exceed 50 when the results are greater than 5x the reporting limit.
- The desired spiking level is 50% of the highest calibration standard. However, the total concentration in the MS (including the MS and native concentration in the unspiked sample) should not exceed 75% of the highest calibration standard in order for a proper evaluation to be performed. The purpose of the matrix spike is to determine whether the sample matrix contributes bias to the analytical results. The corrected concentrations of each analyte within the matrix spiking solution must be within 40 - 140% of the true value. Lower recoveries of n-nonane are permissible but must be noted in the narrative if <30%.

#### MS/MSD Recoveries and Precision Criteria

Sample ID: FA41811-2\_MS/MSD Matrix/Level: Groundwater  
 Sample ID: FA42031-7\_MS/MSD Matrix/Level: Groundwater

List the %Rs, RPD of the compounds which do not meet the QC criteria.

MS OR MSD	COMPOUND	% R	RPD	QC LIMITS	ACTION

**Note:** MS/MSD and RPD within laboratory control limits.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met and/or see below           

No action is taken on MS/MSD results alone to qualify the entire case. However, used informed professional judgment, the data reviewer may use the MS/MSD results in conjunction with other QC criteria and determine the need for some qualification of the data. In those instances where it can be determined that the results of the MS/MSD affect only the sample spiked, the qualification should be limited to this sample alone. However, it may be determined through the MS/MSD results that the laboratory is having a systematic problem in the analysis of one or more analytes, which affects the associated samples.

### 2. MS/MSD – Unspiked Compounds

List the concentrations of the unspiked compounds and determine the % RSDs of these compounds in the unspiked sample, matrix spike, and matrix spike duplicate.

COMPOUND	CONCENTRATION		MSD	%RPD	ACTION
	SAMPLE	MS			

Criteria: None specified, use %RSD  $\leq$  50 as professional judgment.

Actions:

If the % RSD > 50, qualify the results in the spiked sample as estimate (J).

If the % RSD is not calculable (NC) due to nondetect value in the sample, MS, and/or MSD, use professional judgment to qualify sample data.

A separate worksheet should be used for each MS/MSD pair.

All criteria were met   X    
Criteria were not met and/or see below           

### VIII. LABORATORY CONTROL SAMPLE (LCS/LCSD) ANALYSIS

This data is generated to determine accuracy of the analytical method for various matrices.

#### 1. LCS Recoveries Criteria

List the %R of compounds which do not meet the criteria

LCS ID	COMPOUND	% R	QC LIMIT	ACTION
--------	----------	-----	----------	--------

  LCS\_RECOVERY\_WITHIN\_LABORATORY\_CONTROL\_LIMITS.  

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#### Criteria:

- \* Refer to QAPP for specific criteria.
- \* The spike recovery must be between 40% and 140%. Lower recoveries of n-nonane are permissible. If the recovery of n-nonane is <30%, note the nonconformance in the executive narrative. RPD between LCS/LCSD must be < 25%.

#### Actions:

Actions on LCS recovery should be based on both the number of compounds that are outside the %R and RPD criteria and the magnitude of the exceedance of the criteria.

If the %R of the analyte is > UL, qualify all positive results (j) for the affected analyte in the associated samples and accept nondetects.

If the %R of the analyte is < LL, qualify all positive results (j) and reject (R) nondetects for the affected analyte in the associated samples.

If more than half the compounds in the LCS are not within the required recovery criteria, qualify all positive results as (J) and reject nondetects (R) for all target analyte(s) in the associated samples.

#### 2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix (1 per 20 samples per matrix)? Yes or No.

If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected. Discuss the actions below:

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## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met and/or see below           

### IX. FIELD/LABORATORY DUPLICATE PRECISION

Sample IDs:                    -                   

Matrix:            -           

Field/laboratory duplicate samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which measures only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION
No field/laboratory duplicate analyzed with this data package. MS/MSD recoveries RPD used to assess precision. RPD within laboratory and generally acceptable control limits					

#### Criteria:

The project QAPP should be reviewed for project-specific information.  
 RPD  $\pm$  30% for aqueous samples, RPD  $\pm$  50 % for solid samples if results are  $\geq$  SQL.  
 If both samples and duplicate are  $< 5$  SQL, the RPD criteria is doubled.

SQL = soil quantitation limit

#### Actions:

If both the sample and the duplicate results are nondetects (ND), the RPD is not calculable (NC). No action is needed.

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria.

If one sample result is not detected and the other is  $\geq 5$ x the SQL qualify (J/UJ).

**Note:** If SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is  $< 5$ x the SQL, use professional judgment to determine if qualification is appropriate.

All criteria were met   X    
Criteria were not met and/or see below           

## XI. COMPOUND IDENTIFICATION

The compound identification evaluation is to verify that the laboratory correctly identified target analytes as well as tentatively identified compounds (TICs).

1. Verify that the target analytes were within the retention time windows.
  - Retention time windows must be re-established for each Target EPH Analyte each time a new GC column is installed, and must be verified and/or adjusted on a daily basis.
  - The n-nonane (n-C9) peak must be adequately resolved from the solvent front of the chromatographic run.
  - All surrogates must be adequately resolved from the Aliphatic Hydrocarbon and Aromatic Hydrocarbon standards.
  - For the purposes of this method, adequate resolution is assumed to be achieved if the height of the valley between two peaks is less than 25% of the average height of the two peaks.
  - The n-pentane (C5) and MtBE peaks must be adequately resolved from any solvent front that may be present on the FID and PID chromatograms, respectively.

### 1a. Aliphatic hydrocarbons range:

- Determine the total area count for all peaks eluting 0.1 minutes before the retention time (Rt) for n-C9 and 0.01 minutes before the Rt for n-C19.
- Determine the total area count for all peaks eluting 0.01 minutes before the Rt for n-C19 and 0.1 minutes after the Rt for n-C36.

Are the aliphatic hydrocarbons range properly determined?

Yes? or No?

Comments:

### 1b. Aromatic hydrocarbons range:

- Determine the total area count for all peaks eluting 0.1 minutes before the retention time (Rt) for naphthalene and 0.1 minutes after the Rt for benzo(g,h,i)perylene.
- Determine the peak area count for the sample surrogate (OTP) and fractionation surrogate(s). Subtract these values from the collective area count value.

Are the aliphatic hydrocarbons range properly determined?

Yes? or No?

Comments:

# DATA REVIEW WORKSHEETS

All criteria were met   X  

Criteria were not met and/or see below           

2. If target analytes and/or TICs were not correctly identified, request that the laboratory resubmit the corrected data.

3. Breakthrough determination - Each sample (field and QC sample) must be evaluated for potential breakthrough on a sample specific basis by evaluating the % recovery of the fractionation surrogate (2-bromonaphthalene) and on a batch basis by quantifying naphthalene and 2-methylnaphthalene in both the aliphatic and aromatic fractions of the LCS and LCSD. **If either the concentration of naphthalene or 2-methylnaphthalene in the aliphatic fraction exceeds 5% of the total concentration for naphthalene or 2-methylnaphthalene in the LCS or LCSD, fractionation must be repeated on all archived batch extracts.**

**NOTE:** The total concentration of naphthalene or 2-methylnaphthalene in the LCS/LCSD pair includes the summation of the concentration detected in the aliphatic fraction and the concentration detected in the aromatic fraction.

Comments: Concentration in the aliphatic fraction < 5% of the total  
concentration for naphthalene and 2-methylnaphthalene

4. **Fractionation Check Standard** – A fractionation check solution is prepared containing 14 alkanes and 17 PAHs at a nominal concentration of 200 ng/μl of each constituent. The Fractionation Check Solution must be used to evaluate the fractionation efficiency of each new lot of silica gel/cartridges, and establish the optimum hexane volume required to efficiently elute aliphatic hydrocarbons while not allowing significant aromatic hydrocarbon breakthrough. For each analyte contained in the fractionation check solution, excluding n-nonane, the Percent Recovery must be between 40 and 140%. A 30% Recovery is acceptable for n-nonane.

Is a fractionation check standard analyzed?

Yes? or No?

Comments: Not applicable.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met and/or see below           

### XII. QUANTITATION LIMITS AND SAMPLE RESULTS

The sample quantitation evaluation is to verify laboratory quantitation results.

In order to demonstrate the absence of aliphatic mass discrimination, the response ratio of C28 to C20 must be at least 0.85. If <0.85, this nonconformance must be noted in the laboratory case narrative.

The chromatograms of Continuing Calibration Standards for aromatics must be reviewed to ensure that there are no obvious signs of mass discrimination.

Is aliphatic mass discrimination observed in the sample? Yes? or No?

Is aromatic mass discrimination observed in the sample? Yes? or No?

1. In the space below, please show a minimum of one sample calculation:

(C11 – C22, Aromatics)

Computer printout

2. If requested, verify that the results were above the laboratory method detection limit (MDLs).

3. If dilutions performed, were the SQLs elevated accordingly by the laboratory? List the affected samples and dilution factor in the table below.

SAMPLE ID	DILUTION FACTOR	REASON FOR DILUTION

If dilution was not performed, estimate results (J) for the affected compounds. List the affected samples/compounds:

\_\_\_\_\_

\_\_\_\_\_



## EXECUTIVE NARRATIVE

SDG No: **FA41854** Laboratory: **Accutest, Orlando**  
Analysis: **SW846-8081B** Number of Samples: **4**

Location: **BMSMC, Humacao, PR**

SUMMARY: Four (4) samples were analyzed for the TCL pesticides list following method SW846-8081B. The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence *Hazardous Waste Support Section SOP No. HW-36A, Revision 0, June, 2015. SOM02.2. Pesticide Data Validation*. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

Results are valid and can be used for decision making purposes.

Critical issues: **None**  
Major: **None**  
Minor: **None**

Critical findings: **None**  
Major findings: **None**  
Minor findings: **None**

COMMENTS: Results are valid and can be used for decision making purposes.

Reviewers Name: **Rafael Infante**  
**Chemist License 1888**

Signature:

A handwritten signature in blue ink, reading "Rafael Infante", is written over a horizontal line.

Date:

**April 14, 2017**

# ORGANIC DATA SAMPLE SUMMARY

Sample ID: FA41854-1

Sample location: BMSMC, Humacao, PR

Sampling date: 3/6/2017

Matrix: Groundwater

METHOD: 8081B

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Dieldrin	0.010	ug/L	1.0	-	U	Yes

Sample ID: FA41854-2

Sample location: BMSMC, Humacao, PR

Sampling date: 3/6/2017

Matrix: Groundwater

METHOD: 8081B

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Dieldrin	0.010	ug/L	1.0	-	U	Yes

Sample ID: FA41854-2MS

Sample location: BMSMC, Humacao, PR

Sampling date: 3/6/2017

Matrix: Groundwater

METHOD: 8081B

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Dieldrin	0.59	ug/L	1.0	-	-	Yes

Sample ID: FA41854-2MSD

Sample location: BMSMC, Humacao, PR

Sampling date: 3/6/2017

Matrix: Groundwater

METHOD: 8081B

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable
Dieldrin	0.62	ug/L	1.0	-	-	Yes

# DATA REVIEW WORKSHEETS

Project/CasNumber: FA41854  
 Sampling Date: 03/06/2017  
 Shipping Date: 03/07/2017  
 EPA Region No.: 2

## REVIEW OF PESTICIDE ORGANIC PACKAGE

The following guidelines for evaluating volatile organics were created to delineate required validation actions. This document will assist the reviewer in using professional judgment to make more informed decision and in better serving the needs of the data users. The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence *Hazardous Waste Support Section SOP No. HW-36A, Revision 0, June, 2015. SOM02.2. Pesticide Data Validation*. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

The hardcopied (laboratory name) Accutest data package received has been reviewed and the quality control and performance data summarized. The data review for VOCs included:

Lab. Project/SDG No.: FA41854 Sample matrix: Groundwater  
 No. of Samples: 4  
 Trip blank No.: -  
 Field blank No.: -  
 Equipment blank No.: -  
 Field duplicate No.: -  
 Field spikes No.: FA41954-2MS/-2MSD  
 QC audit samples: -

<input checked="" type="checkbox"/> Data Completeness	<input checked="" type="checkbox"/> Laboratory Control Spikes
<input checked="" type="checkbox"/> Holding Times	<input checked="" type="checkbox"/> Field Duplicates
<input type="checkbox"/> GC/MS Tuning	<input checked="" type="checkbox"/> Calibrations
<input checked="" type="checkbox"/> Internal Standard Performance	<input checked="" type="checkbox"/> Compound Identifications
<input checked="" type="checkbox"/> Blanks	<input checked="" type="checkbox"/> Compound Quantitation
<input checked="" type="checkbox"/> Surrogate Recoveries	<input checked="" type="checkbox"/> Quantitation Limits
<input checked="" type="checkbox"/> Matrix Spike/Matrix Spike Duplicate	

Overall Comments: Dieldrin by SW846-8081B  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

### Definition of Qualifiers:

J- Estimated results	U- Compound not detected
R- Rejected data	UJ- Estimated nondetect

Reviewer: Rafael Difant  
 Date: April 14, 2017



## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met  
and/or see below \_\_\_\_\_

### HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE EXTRACTED/ANALYZED	ACTION
Samples properly preserved. All samples extracted and analyzed within the required criteria.			

#### Note:

#### Criteria

Aqueous samples - seven (7) days from sample collection for extraction; 40 days from sample collection for analysis.

Non-aqueous samples – fourteen (14) days from sample collection for extraction; 40 days from sample collection for analysis.

Cooler temperature (Criteria:  $4 \pm 2$  °C): 3.0/3.2 °C - OK

#### Actions

**Qualify aqueous sample results using preservation and technical holding time information as follows:**

- If there is no evidence that the samples were properly preserved ( $T = 4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ ), and the samples were extracted or analyzed within the technical holding times, qualify detects as estimated (J) and non-detects as estimated (UJ).
- If there is no evidence that the samples were properly preserved ( $T = 4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ ), and the samples were extracted or analyzed outside the technical holding times, qualify detects as estimated (J) and non-detects as estimated (UJ).
- If the samples were properly preserved, and were extracted and analyzed within the technical holding times, no qualification of the data is necessary.
- If the samples were properly preserved, and were extracted or analyzed outside the technical holding times, qualify detects as estimated (J) and non-detects as estimated (UJ). Note in the Data Review Narrative that holding times were exceeded and the effect of exceeding the holding time on the resulting data.

## DATA REVIEW WORKSHEETS

- e. Use professional judgment to qualify samples whose temperature upon receipt at the laboratory is either below 2 degrees centigrade or above 6 degrees centigrade.
- f. If technical holding times are grossly exceeded, use professional judgment to qualify the data.

### **Qualify non-aqueous sample results using preservation and technical holding time information as follows:**

- a. If there is no evidence that the samples were properly preserved ( $T = 4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ ), and the samples were extracted or analyzed within the technical holding time, qualify detects as estimated (J) and non-detects as estimated (UJ).
- b. If there is no evidence that the samples were properly preserved ( $T = 4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ ), and the samples were extracted or analyzed outside the technical holding time, qualify detects as estimated (J) and non-detects as estimated (UJ).
- c. If the samples were properly preserved, and were extracted and analyzed within the technical holding time, no qualification of the data is necessary.
- d. If the samples were properly preserved, and were extracted or analyzed outside the technical holding time, qualify detects as estimated (J) and non-detects as estimated (UJ). Note in the Data Review Narrative that holding times were exceeded and the effect of exceeding the holding time on the resulting data.
- e. Use professional judgment to qualify samples whose temperature upon receipt at the laboratory is either below 2 degrees centigrade or above 6 degrees centigrade.
- f. If technical holding times are grossly exceeded, use professional judgment to qualify the data.

## DATA REVIEW WORKSHEETS

All criteria were met X  
Criteria were not met see below       

### GAS CHROMATOGRAPH WITH ELECTRON CAPTURE DETECTOR (GC/ECD) INSTRUMENT PERFORMANCE CHECK (SECTIONS 1 TO 5)

#### 1. Resolution Check Mixture

##### Criteria

Is the resolution between two adjacent peaks in the Resolution Check Mixture C greater than or equal to 80.0% for all analytes for the primary column and greater than or equal to 50.0% for the confirmation column? Yes? or No?

Is the resolution between two adjacent peaks in the Resolution Check Mixture (A and B) greater than or equal to 60.0%? Yes? or No?

Note: If resolution criteria are not met, the quantitative results may not be accurate due to inadequate resolution. Qualitative identifications may also be questionable if coelution exists.

##### Action

- a. Qualify detects for target compounds that were not adequately resolved as tentatively identified (NJ).
- b. Qualify non-detected compounds as unusable (R).

#### 2. Performance Evaluation Mixture (PEM) Resolution Criteria

##### Criteria

Is PEM analysis performed at the required frequency (at the end of each pesticide initial calibration sequence and every 12 hours)? Yes? or No?

##### Action

- a. If PEM is not performed at the required frequency, qualify all associated sample and blank results as unusable (R).

##### Criteria

Is PEM % Resolution < 90%? Yes? or No?

##### Action

- a. a. Qualify detects for target compounds that were not adequately resolved as tentatively identified (NJ).
- b. Qualify non-detected compounds as unusable (R).



## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met see below       

### 3. PEM 4,4'-DDT Breakdown

#### Criteria

Is the PEM 4,4'-DDT % Breakdown >20.0% and 4,4'-DDT is detected? Yes? or No?

#### Action

a. Qualify detects for 4,4'-DDT; detects for 4,4'-DDD; and detects for 4,4'-DDE as estimated (J)

#### Criteria

Is the PEM 4,4'-DDT % Breakdown >20.0% and 4,4'-DDT is not detected Yes? or No?

#### Action

- a. Qualify non-detects for 4,4'- DDT as unusable (R )
- b. Qualify detects for 4,4'-DDD as tentatively identified (NJ)
- c. Qualify detects for 4,4'-DDE as tentatively identified (NJ)

### 4. PEM Endrin Breakdown

#### Criteria

Is the PEM Endrin % Breakdown >20.0% and Endrin is detected? Yes? or No?

#### Action

a. Qualify detects for Endrin; detects for Endrin aldehyde; and detects for Endrin ketone as estimated (J)

#### Criteria

Is the PEM Endrin % Breakdown >20.0% and Endrin is not detected Yes? or No?

#### Action

- a. Qualify non-detects for Endrin as unusable (R )
- b. Qualify detects for Endrin aldehyde as tentatively identified (NJ)
- c. Qualify detects for Endrin ketone as tentatively identified (NJ)

## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met see below       

### 5. Mid-point Individual Standard Mixture Resolution -

#### Criteria

Is the resolution between two adjacent peaks in the Resolution Check Mixture C greater than or equal to 80.0% for all analytes for the primary column and greater than or equal to 50.0% for the confirmation column? Yes? or No?

Is the resolution between two adjacent peaks in the Resolution Check Mixture (A and B) greater than or equal to 90.0%? Yes? or No?

Note: If resolution criteria are not met, the quantitative results may not be accurate due to inadequate resolution. Qualitative identifications may also be questionable if coelution exists.

#### Action

- a. Qualify detects for target compounds that were not adequately resolved as tentatively identified (NJ).
- b. Qualify non-detected compounds as unusable (R).

#### Criteria

Is mid-point individual standard mixture analysis performed at the required frequency (every 12 hours)? Yes? or No?

#### Action

- a. If the mid-point individual standard mixture analysis is not performed at the required frequency, qualify all associated sample and blank results as unusable (R).

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met  
 and/or see below           

### CALIBRATION VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration: 03/10/17  
 Dates of initial calibration verification: 03/10/17  
 Dates of continuing calibration: 03/17/17  
 Dates of final calibration: 03/17/17  
 Instrument ID numbers: ECD\_5  
 Matrix/Level: Aqueous/low

DATE	LAB ID#	FILE	CRITERIA OUT RFs, %RSD, %D, r	COMPOUND	SAMPLES AFFECTED

**Note:** Initial and initial calibration verification within the guidance document performance criteria. Continuing calibration % differences meet the performance criteria in the two columns.

Final calibration verification included in data package. No action taken.

#### Criteria

Are a five point calibration curve delivered with concentration levels as shown in Table 3 of SOP HW-36A, Revision 0, June, 2015? Yes? or No?

#### Actions

If the standard concentrations listed in Table 3 are not used, use professional judgment to evaluate the effect on the data

#### Criteria

Are RT Windows calculated correctly? Yes? or No?

#### Action

Recalculate the windows and use the corrected values for all evaluations.

#### Criteria

Are the Percent Relative Standard Deviation (%RSD) of the CFs for each of the single component target compounds less than or equal to 20.0%, except for alpha-BHC and delta-BHC?

Yes? or No?

## DATA REVIEW WORKSHEETS

All criteria were met X  
Criteria were not met \_\_\_\_\_  
and/or see below \_\_\_\_\_

Are the %RSD of the CFs for alpha-BHC and delta-BHC less than or equal to 25.0%. Yes? or No?

Is the %RSD of the CFs for each of the Toxaphene peaks must be < 30% when 5-point ICAL is performed? Yes? or No?

Is the %RSD of the CFs for the two surrogates (tetrachloro-m-xylene and decachlorobiphenyl) less than or equal to 30.0%. Yes? or No?

### Action

- If the %RSD criteria are not met, qualify detects as estimated (J) and use professional judgment to qualify non-detected target compounds.
- If the %RSD criteria are within allowable limits, no qualification of the data is necessary

### Continuing Calibration Checks

#### Criteria

Is the continuing calibration standard analyzed at the acceptable time intervals? Yes? or No?

#### Action

- If more than 14 hours has elapsed from the injection of the instrument blank that begins an analytical sequence (opening CCV) and the injection of either a PEM or mid-point concentration of the Individual Standard Mixtures (A and B) or (C), qualify all data as unusable (R).
- If more than 12 hours has elapsed from the injection of the instrument blank that begins an analytical sequence (opening CCV) and the injection of the last sample or blank that is part of the same analytical sequence, qualify all data as unusable (R).
- If more than 72 hours has elapsed from the injection of the sample with a Toxaphene detection and the Toxaphene Calibration Verification Standard (CS3), qualify all data as unusable (R).

#### Criteria

Is the Percent Difference (%D) within  $\pm 25.0\%$  for the PEM sample? Yes? or No?

#### Action

- Qualify associated detects as estimated (J) and non-detects as estimated (UJ).

#### Criteria

For the Calibration Verification Standard (CS3); is the Percent Difference (%D) within  $\pm 25.0\%$ ? Yes? or No?

#### Action

Qualify associated detects as estimated (J) and non-detects as estimated (UJ).

## DATA REVIEW WORKSHEETS

### Criteria

Is the PEM 4,4'-DDT % Breakdown >20.0% and 4,4'-DDT is detected? Yes? or No?

### Action

- a. Qualify detects for 4,4'-DDT; detects for 4,4'-DDD; and detects for 4,4'-DDE as estimated (J)
- b. Non-detected associated compounds are not qualified

### Criteria

Is the PEM 4,4'-DDT % Breakdown >20.0% and 4,4'-DDT is not detected? Yes? or No?

### Action

- a. Qualify non-detects for 4,4'- DDT as unusable (R )
- b. Qualify detects for 4,4'-DDD as tentatively identified (NJ)
- c. Qualify detects for 4,4'-DDE as tentatively identified (NJ)

### Criteria

Is the PEM Endrin % Breakdown >20.0% and Endrin is detected? Yes? or No?

### Action

- a. Qualify detects for Endrin; detects for Endrin aldehyde; and detects for Endrin ketone as estimated (J)
- b. Non-detected associated compounds are not qualified

### Criteria

Is the PEM Endrin % Breakdown >20.0% and Endrin is not detected? Yes? or No?

### Action

- a. Qualify non-detects for Endrin as unusable (R )
- b. Qualify detects for Endrin aldehyde as tentatively identified (NJ)
- c. Qualify detects for Endrin ketone as tentatively identified (NJ)

A separate worksheet should be filled for each initial curve

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met  
 and/or see below \_\_\_\_\_

### BLANK ANALYSIS RESULTS (Sections 1 & 2)

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

List the contamination in the blanks below. High and low levels blanks must be treated separately.

CRQL concentration \_\_\_\_\_ N/A \_\_\_\_\_

#### Laboratory blanks

DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
_No target analytes detected in method blanks at a reporting limit of 0.01, 0.02, and 0.25_				
_ug/L_____				
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

#### Field/Equipment/Trip blank

DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
_No trip/field/equipment blanks analyzed with this data package._____				
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

## DATA REVIEW WORKSHEETS

All criteria were met ☒   
 Criteria were not met   
 and/or see below \_\_\_\_\_

### BLANK ANALYSIS RESULTS (Section 3)

#### Blank Actions

Action Levels (ALs) should be based upon the highest concentration of contaminant determined in any blank. Do not qualify any blank with another blank. The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. No positive sample results should be reported unless the concentration of the compound in the samples exceeds the ALs:

The concentration of non-target compounds in all blanks must be less than or equal to 10 µg/L. The concentration of each target compound found in the method or field blanks must be less than its CRQL listed in the method.

Data concerning the field blanks are not evaluated as part of the CCS process. If field blanks are present, the data reviewer should evaluate this data in a similar fashion as the method blanks.

Specific actions are as follows:

#### Blank Actions for Pesticide Analyses

Blank Type	Blank Result	Sample Result	Action for Samples
Method, Sulfur Cleanup, Instrument, Field, TCLP/SPLP	Detects	Not detected	No qualification required
	< CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL	No qualification required
	> CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL and ≤ blank concentration	Report blank value for sample concentration with a U
		≥ CRQL and > blank concentration	No qualification required
	= CRQL	≤ CRQL	Report CRQL value with a U
		> CRQL	No qualification required
	Gross contamination	Detects	Report blank value for sample concentration with a U

## DATA REVIEW WORKSHEETS

All criteria were met X  
Criteria were not met  
and/or see below \_\_\_\_\_

[illegible]



## DATA REVIEW WORKSHEETS

All criteria were met X  
 Criteria were not met  
 and/or see below \_\_\_\_\_

### SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery.

Matrix: Aqueous/Solid

Lab Sample ID	Lab File ID	S1 a	S2 a
FA41854-1	KK82213.D	105	37
FA41854-2	KK82216.D	107	57
OP64131-BS	KK82206.D	109	104
OP64131-BS2	KK82207.D	101	89
OP64131-MB	KK82208.D	105	105
OP64131-MS	KK82217.D	114	118
OP64131-MSD	KK82218.D	118	110

Surrogate Compounds	Recovery Limits (Aqueous)
S1 = Tetrachloro-m-xylene	42-127%
S2 = Decachlorobiphenyl	27-127%

(a) Recovery from GC signal #1

**Note:** Surrogate recoveries were within laboratory control limits.

**Actions:**

- For any surrogate recovery greater than 150%, qualify detected target compounds as biased high (J+).
- Do not qualify non-detected target compounds for surrogate recovery > 150 %.
- If both surrogate recoveries are greater than or equal to 30% and less than or equal to 150%, no qualification of the data is necessary.
- For any surrogate recovery greater than or equal to 10% and less than 30%, qualify detected target compounds as biased low (J-).
- For any surrogate recovery greater than or equal to 10% and less than 30%, qualify non-detected target compounds as approximated (UJ).

## DATA REVIEW WORKSHEETS

f. If low surrogate recoveries are from sample dilution, professional judgment should be used to determine if the resulting data should be qualified. If sample dilution is not a factor:

- i. Qualify detected target compounds as biased low (J-).
- ii. Qualify non-detected target compounds as unusable (R).

g. If surrogate RTs in PEMs, Individual Standard Mixtures, samples, and blanks are outside of the RT Windows, the reviewer must use professional judgment to qualify data.

h. If surrogate RTs are within RT windows, no qualification of the data is necessary.

i. If the two surrogates were not added to all samples, MS/MSDs, standards, LCSs, and blanks, use professional judgment in qualifying data as missing surrogate analyte may not directly apply to target analytes.

### Summary Surrogate Actions for Pesticide Analyses

Criteria	Action*	
	Detected Target Compounds	Non-detected Target Compounds
%R > 150%	J+	No qualification
30% < %R < 150%	No qualification	
10% < %R < 30%	J-	UJ
%R < 10% (sample dilution not a factor)	J-	R
%R < 10% (sample dilution is a factor)	Use professional judgment	
RT out of RT window	Use professional judgment	
RT within RT window	No qualification	

\* Use professional judgment in qualifying data, as surrogate recovery problems may not directly apply to target analytes.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met  
 and/or see below \_\_\_\_\_

### MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples. If any % R in the MS or MSD falls outside the designated range, the reviewer should determine if there are matrix effects, i.e. LCS data are within the QC limits but MS/MSD data are outside QC limit.

#### 1. MS/MSD Recoveries and Precision Criteria

Data for MS and MSDs will not be present unless requested by the Region.

Notify the Contract Laboratory Program Project Officer (CLP PO) if a field blank was used for the MS and MSD, unless designated as such by the Region.

**NOTE:** For a Matrix Spike that does not meet criteria, apply the action to only the field sample used to prepare the Matrix Spike sample. If it is clearly stated in the data validation materials that the samples were taken through incremental sampling or some other method guaranteeing the homogeneity of the sample group, then the entire sample group may be qualified.

List the %Rs, RPD of the compounds which do not meet the criteria.

Sample ID: FA41854-2MS/2MSD

Matrix/Level: Aqueous

Sample ID: FA41811-2MS/2MSD

Matrix/Level: Aqueous

Sample ID: FA42031-7MS/7MSD

Matrix/Level: Aqueous

The QC reported here applies to the following samples:  
 FA41854-1, FA41854-2

Method: SW846 8081B

Compound	ug/l	Q	Spike ug/l	MS ug/l	MS %	Spike ug/l	MSD ug/l	MSD %	RPD	Limits Rec/RPD
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**Note:** MS/MSD % recoveries and RPD within laboratory control limits except for the cases described in this document. Results apply to unspiked sample. Unspiked sample was from another job. No qualifications made.

#### Action

No qualification of the data is necessary on MS and MSD data alone. However, using professional judgment, the validator may use the MS and MSD results in conjunction with other QC criteria and determine the need for some qualification of the data.

A separate worksheet should be used for each MS/MSD pair.

## DATA REVIEW WORKSHEETS

All criteria were met X  
 Criteria were not met  
 and/or see below \_\_\_\_\_

### LABORATORY CONTROL SAMPLE (LCS) ANALYSIS

This data is generated to determine accuracy of the analytical method for various matrices.

#### 1. LCS Recoveries Criteria

LCS Spike Compound	Recovery Limits (%)
gamma-BHC	50 – 120
Heptachlor epoxide	50 – 150
Dieldrin	30 – 130
4,4'-DDE	50 – 150
Endrin	50 – 120
Endosulfan sulfate	50 – 120
trans-Chlordane	30 – 130
Tetrachloro-m-xylene (surrogate)	30 – 150
Decachlorobiphenyl (surrogate)	30 – 150

LCS concentrations: 0.25 ug/l

List the %R of compounds which do not meet the criteria

LCS ID	COMPOUND	% R	QC LIMIT
_____ %_recovery_and_RPD_within_laboratory_control_limits. _____			
_____			
_____			

#### Note:

#### Action

The following guidance is suggested for qualifying sample data for which the associated LCS does not meet the required criteria.

- If the LCS recovery exceeds the upper acceptance limit, qualify detected target compounds as estimated (J). Do not qualify non-detected target compounds.
- If the LCS recovery is less than the lower acceptance limit, qualify detected target compounds as estimated (J) and non-detects as unusable (R).
- Use professional judgment to qualify data for compounds other than those compounds that are included in the LCS.
- Use professional judgment to qualify non-LCS compounds. Take into account the compound class, compound recovery efficiency, analytical problems associated with each compound, and comparability in the performance of the LCS compound to the non-LCS compound.
- If the LCS recovery is within allowable limits, no qualification of the data is necessary.

## DATA REVIEW WORKSHEETS

### 2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix? **Yes** or No.

If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected.

## DATA REVIEW WORKSHEETS

All criteria were met \_\_\_\_\_  
Criteria were not met \_\_\_\_\_  
and/or see below \_\_N/A\_\_\_\_

### FLORISIL CARTRIDGE PERFORMANCE CHECK

NOTE: Florisil cartridge cleanup is mandatory for all extracts.

#### Criteria

Is the Florisil cartridge performance check conducted at least once on each lot of cartridges used for sample cleanup or every 6 months, whichever is most frequent? Yes? or No? **N/A**

#### Criteria

Are the results for the Florisil Cartridge Performance Check solution included with the data package? Yes? or No? **N/A**

Note: If % criteria are not met, examine the raw data for the presence of polar interferences and use professional judgment in qualifying the data as follows:

#### Action:

- a. If the Percent Recovery is greater than 120% for any of the pesticide target compounds in the Florisil Cartridge Performance Check, qualify detected compounds as estimated (J). Do not qualify non-detected target compounds.
- b. If the Percent Recovery is greater than or equal to 80% and less than or equal to 120% for all the pesticide target compounds, no qualification of the data is necessary.
- c. If the Percent Recovery is greater than or equal to 10% and less than 80% for any of the pesticide target compounds in the Florisil Cartridge Performance Check, qualify detected target compounds as estimated (J) and non-detected target compounds as approximated (UJ).
- d. If the Percent Recovery is less than 10% for any of the pesticide target compounds in the Florisil Cartridge Performance Check, qualify detected compounds as estimated (J) and qualify non-detected target compounds as unusable (R).
- e. If the Percent Recovery of 2,4,5-trichlorophenol in the Florisil Cartridge Performance Check is greater than or equal to 5%, use professional judgment to qualify detected and non-detected target compounds, considering interference on the sample chromatogram.

Note: State in the Data Review Narrative potential effects on the sample data resulting from the Florisil Cartridge Performance Check analysis not yielding acceptable results.

Note:    No information for Florisil cartridge performance check included in data package.

## DATA REVIEW WORKSHEETS

All criteria were met \_\_\_\_\_  
Criteria were not met \_\_\_\_\_  
and/or see below \_\_\_\_\_

### GEL PERMEATION CHROMATOGRAPHY (GPC) PERFORMANCE CHECK

NOTE: GPC cleanup is mandatory for all soil samples.

If GPC criteria are not met, examine the raw data for the presence of high molecular weight contaminants; examine subsequent sample data for unusual peaks; and use professional judgment in qualifying the data. Notify the Contract Laboratory Program Project Officer (CLP PO) if the laboratory chooses to analyze samples under unacceptable GPC criteria.

#### Action:

- a. If the Percent Recovery is less than 10% for the pesticide compounds and surrogates during the GPC calibration check, the non-detected target compounds may be suspect, qualify detected compounds as estimated (J).
- b. If the Percent Recovery is less than 10% for the pesticide compounds and surrogates during the GPC calibration check, qualify all non-detected target compounds as unusable (R).
- c. If the Percent Recovery is greater than or equal to 10% and is less than 80% for any of the pesticide target compounds in the GPC calibration, qualify detected target compounds as estimated (J) and non-detected target compounds as approximated (UJ).
- d. If the Percent Recovery is greater than or equal to 80% and less than or equal to 120% for all the pesticide target compounds, no qualification of the data is necessary.
- e. If high recoveries (i.e., greater than 120%) were obtained for the pesticides and surrogates during the GPC calibration check, qualify detected compounds as estimated (J). Do not qualify non-detected target compounds.

Note: State in the Data Review Narrative potential effects on the sample data resulting from the GPC cleanup analyses not yielding acceptable results.

**Note:** No information for performance of GPC cleanup included in data package.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met  
and/or see below           

### TARGET COMPOUND IDENTIFICATION

#### Criteria:

1. Is Retention Times (RTs) of both of the surrogates and reported target compounds in each sample within the calculated RT Windows on both columns? Yes? or No?

2. Is the Tetrachloro-m-xylene (TCX) RT  $\pm 0.05$  minutes of the Mean RT (RT) determined from the initial calibration and Decachlorobiphenyl (DCB) within  $\pm 0.10$  minutes of the RT determined from the initial calibration? Yes? or No?

3. Is the Percent Difference (%D) for the detected mean concentrations of a pesticide target compound between the two Gas Chromatograph (GC) columns within the inclusive range of  $\pm 25.0$  %? Yes? or No?

4. When no analytes are identified in a sample; are the chromatograms from the analyses of the sample extract and the low-point standard of the initial calibration associated with those analyses on the same scaling factor? Yes? or No?

5. Does the chromatograms display the Single Component Pesticides (SCPs) detected in the sample and the largest peak of any multi-component analyte detected in the sample at less than full scale. Yes? or No?

6. If an extract is diluted; does the chromatogram display SCPs peaks between 10-100% of full scale, and multi-component analytes between 25-100% of full scale? Yes? or No? N/A

7. For any sample; does the baseline of the chromatogram return to below 50% of full scale before the elution time of alpha-BHC, and also return to below 25% of full scale after the elution time of alpha-BHC and before the elution time of DCB? Yes? or No?

8. If a chromatogram is replotted electronically to meet these requirements; is the scaling factor used displayed on the chromatogram, and both the initial chromatogram and the replotted chromatogram submitted in the data package. Yes? or No?

#### Action:

a. If the qualitative criteria for both columns were not met, all target compounds that are reported as detected should be considered non-detected.

b. Use professional judgment to assign an appropriate quantitation limit using the following guidance:

- i. If the detected target compound peak was sufficiently outside the pesticide RT Window, the reported values may be a false positive and should be replaced with the sample Contract Required Quantitation Limits (CRQL) value.



## DATA REVIEW WORKSHEETS

- ii. If the detected target compound peak poses an interference with potential detection of another target peak, the reported value should be considered and qualified as unusable (R).
- c. If the data reviewer identifies a peak in both GC column analyses that falls within the appropriate RT Windows, but was reported as a non-detect, the compound may be a false negative. Use professional judgment to decide if the compound should be included.

Note: State in the Data Review Narrative all conclusions made regarding target compound identification.

- d. If the Toxaphene peak RT windows determined from the calibration overlap with SCPs or chromatographic interferences, use professional judgment to qualify the data.
- e. If target compounds were detected on both GC columns, and the Percent Difference between the two results is greater than 25.0%, consider the potential for coelution and use professional judgment to decide whether a much larger concentration obtained on one column versus the other indicates the presence of an interfering compound. If an interfering compound is indicated, use professional judgment to determine how best to report, and if necessary, qualify the data according to these guidelines.
- f. If Toxaphene exhibits a marginal pattern-matching quality, use professional judgment to establish whether the differences are due to environmental "weathering" (i.e., degradation of the earlier eluting peaks relative to the later eluting peaks). If the presence of Toxaphene is strongly suggested, report results as presumptively present (N).

## GAS CHROMATOGRAPH/MASS SPECTROMETER (GC/MS) CONFIRMATION

NOTE: This confirmation is not usually provided by the laboratory. In cases where it is provided, use professional judgment to determine if data qualified with "C" can be salvaged if it was previously qualified as unusable (R).

### Action:

- a. If the quantitative criteria for both columns were met ( $\geq 5.0$  ng/ $\mu$ L for SCPs and  $\geq 125$  ng/ $\mu$ L for Toxaphene), determine whether GC/MS confirmation was performed. If it was performed, qualify the data using the following guidance:
  - i. If GC/MS confirmation was not required because the quantitative criteria for both columns was not met, but it was still performed, use professional judgment when evaluating the data to decide whether the detect should be qualified with "C".
  - ii. If GC/MS confirmation was performed, but unsuccessful for a target compound detected by GC/ECD analysis, qualify those detects as "X".

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met  
 and/or see below           

### COMPOUND QUANTITATION AND REPORTED CONTRACT REQUIRED QUANTITATION LIMITS (CRQLS)

The sample quantitation evaluation is to verify laboratory quantitation results. In the space below, please show a minimum of one sample calculation:

FA41854-2MS

Dieldrin

RF =  $5.754 \times 10^4$

$$\begin{aligned} [ ] &= (3381185) / (5.754 \times 10^4) \\ &= 58.76 \text{ ppb} \quad \text{Ok} \end{aligned}$$

#### Note:

#### Action:

- If sample quantitation is different from the reported value, qualify result as unusable (R).
- When a sample is analyzed at more than one dilution, the lowest CRQLs are used unless a QC exceedance dictates the use of the higher CRQLs from the diluted sample.
- Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and its corresponding value on the original reporting form and substituting the data from the diluted sample.
- Results between the MDL and CRQL should be qualified as estimated (J).
- Results less than the MDL should be reported at the CRQL and qualified (U). MDLs themselves are not reported.
- For non-aqueous samples, if the percent moisture is less than 70.0%, no qualification of the data is necessary. If the percent moisture is greater than or equal to 70.0% and less than 90.0%, qualify detects as estimated (J) and non-detects as approximated (UJ). If the percent moisture is greater than or equal to 90.0%, qualify detects as estimated (J) and non-detects as unusable (R) (see Table).

#### Percent Moisture Actions for Pesticide Analysis for Non-Aqueous Samples

Criteria	Action	
	Detected Associated Compounds	Non-detected Associated Compounds
% Moisture < 70.0	No qualification	
70.0 < % Moisture < 90.0	J	UJ
% Moisture > 90.0	J	R

## DATA REVIEW WORKSHEETS

List samples which have  $\leq 50\%$  solids

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Note: If any discrepancies are found, the Region's designated representative may contact the laboratory to obtain additional information that could resolve any differences. If a discrepancy remains unresolved, the reviewer must use professional judgment to decide which value is the most accurate. Under these circumstances, the reviewer may determine that qualification of data is warranted. Note in the Data Review Narrative a description of the reasons for data qualification and the qualification that is applied to the data.

Dilution performed

SAMPLE ID	DILUTION FACTOR	REASON FOR DILUTION

## DATA REVIEW WORKSHEETS

All criteria were met \_\_N/A\_\_  
Criteria were not met  
and/or see below \_\_\_\_\_

### FIELD DUPLICATE PRECISION

**NOTE:** In the absence of QAPP guidance for validating data from field duplicates, the following action will be taken.

Field duplicates samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples. Identify which samples within the data package are field duplicates. Estimate the relative percent difference (RPD) between the values for each compound. If large RPDs (> 50%) is observed, confirm identification of samples and note difference in the executive summary.

Sample IDs: \_\_\_\_\_ - \_\_\_\_\_

Matrix: \_\_\_\_\_ - \_\_\_\_\_

COMPOUND	SQL ug/L	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION
No field/laboratory duplicate analyzed with this data package. MS/MSD % recovery RPD used to assess precision. RPD within the required criteria of < 50 %.					

#### Actions:

a. Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria. For organics, only the sample and duplicate will be qualified.

b. If an RPD cannot be calculated because one or both of the sample results is not detected, the following actions apply:

- i. If one sample result is not detected and the other is greater than 5x the SQL qualify (J/UJ).
- ii. If one sample value is not detected and the other is greater than 5x the SQL and the SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.
- iii. If one sample value is not detected and the other is less than 5x, use professional judgment to determine if qualification is appropriate.
- iv. If both sample and duplicate results are not detected, no action is needed.

## DATA REVIEW WORKSHEETS

### OVERALL ASSESSMENT OF DATA

#### Action:

1. Use professional judgment to determine if there is any need to qualify data which were not qualified based on the Quality Control (QC) criteria previously discussed.
2. Write a brief narrative to give the user an indication of the analytical limitations of the data.

**Note:** The Contract Laboratory Program Project Officer (CLP PO) must be informed if any inconsistency of the data with the Sample Delivery Group (SDG) Narrative. If sufficient information on the intended use and required quality of the data is available, the reviewer should include their assessment of the usability of the data within the given context. This may be used as part of a formal Data Quality Assessment (DQA).

**Overall assessment of the data:** Results are valid; the data can be used for decision making purposes.